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TITANIUM SURFACE TREATMENTS FOR ADHESIVE BONDING

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The aircraft industry prepares titanium surfaces for adhesive bonding using various types of abrasive, etchant, chemical and anodize treatments. A study was made to determine pretreatment effects on adhesive bond durability of titanium under severe environmental conditions of temperature, humidity and stress. Replicate sets of Ti-6Al-4V titanium specimens were provided to aircraft manufacturers who prepared the sets using eleven different pretreatment processes. The manufacturers also bonded the specimens with the same four adhesive systems. Wedge specimens, bonded from 0.150-inch thick Ti-6Al-4V titanium were

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tested in a 140 F, 100% relative humidity atmosphere for a period of eight weeks. Chromic acid anodize, alkaline etch, chromate-fluoride and alkaline peroxide treatments resulted in substantially lower crack growth rates than phosphate-fluoride treatments. There was a slight difference in performance ranking of bonding pretreatments depending upon the adhesive system used.

SUMMARY

INTRODUCTION

Effective surface pretreatments are necessary to prevent premature failure of adhesive bonded titanium in naval aircraft and missiles. Abrasive, etchant, chemical and anodize methods are used by the aircraft industry to prepare titanium surfaces for adhesive bonding, but the durability has not been established for the different pretreatment processes. A crack extension wedge test study was made to determine pretreatment effects on the durability of adhesively bonded titanium.

RESULTS

Eleven titanium pretreatment processes were evaluated for adhesive bond durability using the wedge test method. Four film adhesives with primers, FM300K/B2127, FM73M/BR127, M329/M329 Type II and EA9628H/BR127, were used to bond the wedge test specimens made from 0.150-inch Ti-6Al-4V titanium sheet. Specimens were exposed in a 140 F, 100% relative humidity (condensing) environment and crack growth measurements were taken at intervals during the 56 day exposure period. The average crack opening measurement ($a+\Delta a$) for each pretreatment after 56 days was as follows:

ID	Pretreatment	Average Crack Opening ($a+\Delta a$), inches
CA5-4	5 volt chromic acid anodize with fluoride	2.44
TU-8	Turco 5578	2.47
CA10-4	10 volt chromic acid anodize with fluoride	2.48
LP-6	Pasa Jell 107C - liquid hone	2.50
AP-9	Alkaline peroxide	2.55
DA-5	Dapcotreat 4023/4000	2.71
DP-2	Pasa Jell 107M - dry hone	2.77
PF-3	Phosphate fluoride with HNO_3 predip	3.98
PF-4	Phosphate fluoride	4.47
VA-7	VAST	4.72
PF-1	Phosphate fluoride - PA modified	4.89

Crack growth during exposure testing (Δa) was predominately of the adhesive failure mode at the interface between the titanium and the adhesive/primer layer.

CONCLUSIONS

The most durable titanium pretreatment systems as determined by wedge crack extension testing were chromic acid anodize with fluoride (both 5 and 10 volt), Turco 5578 alkaline etch, liquid hone Pasa Jell 107C and alkaline peroxide.

Dapcotreat 4000 and dry hone Pasa Jell 107M were slightly lower in overall performance than the above five pretreatments.

The three phosphate fluoride pretreatments along with the VAST pretreatment

resulted in significantly longer crack growth lengths and poorer durability than the other pretreatments.

When using wedge tests, total wedge crack openings ($a+\Delta a$) are preferred over crack growth lengths (Δa) as a measure of durability.

RECOMMENDATIONS

Pretreatment methods recommended for adhesive bonding of titanium are chromic acid anodize with fluoride, Turco 5578, Pasa Jell 107, alkaline peroxide or Dapcotreat 4000. Production experience is limited with the chromic acid anodize and alkaline peroxide processes. Alkaline peroxide solutions are unstable so stringent production controls must be used with this method.

Phosphate fluoride and VAST pretreatments should not be used where high durability titanium adhesive bonds are required.

FUTURE PLANS

The alkaline peroxide process will be developed to improve both stability and determine effective operating ranges. Advantages of the alkaline peroxide method include: the bath does not contain either chromate or fluoride ions, anodizing equipment is not required, operating conditions are reasonable for production, and the treatment provides a stable, bondable surface.

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B A C K G R O U N D

Adhesive bonding is widely used to improve design and reduce costs when joining titanium components in advanced aircraft and missiles. The bonding process is especially applicable to joining fiberglass/epoxy or graphite/epoxy composite materials with titanium since it eliminates or minimizes mechanical fasteners. Adhesive bonded titanium joints, however, are quite susceptible to severe environmental conditions encountered by naval aircraft. Most bond failures originate at the titanium surface illustrating the need for effective pretreatment prior to adhesive bonding. A number of pretreatments have been developed for titanium, but the airframe adhesive bonding industry has not determined which pretreatment processes give the most durable adhesive bonds.

The Naval Air Systems Command (NAVAIR) (AIR-5304) undertook a program of engineering and scientific studies to select the most effective pretreatment process for adhesive bonding of titanium. The Naval Air Development Center (NAVAIRDEVCEN) (Code 6062) managed the program and conducted the crack extension wedge test studies under AIRTASK No. WF61-542-001, Work Unit No. ZM520 (reference (a)). U.S. Army Armament Research and Development Command (ARRADCOM), Dover, New Jersey, provided all titanium specimens and conducted the sustained load stress durability program under reference (b). Martin Marietta Laboratories, Baltimore, Maryland, studied effects on bond durability of titanium pretreatment characteristics and surface reactions under reference (c). Nine airframe manufacturers and three adhesive suppliers also contributed to this program. Program participants, along with their principle functions, are shown in Figure 1.

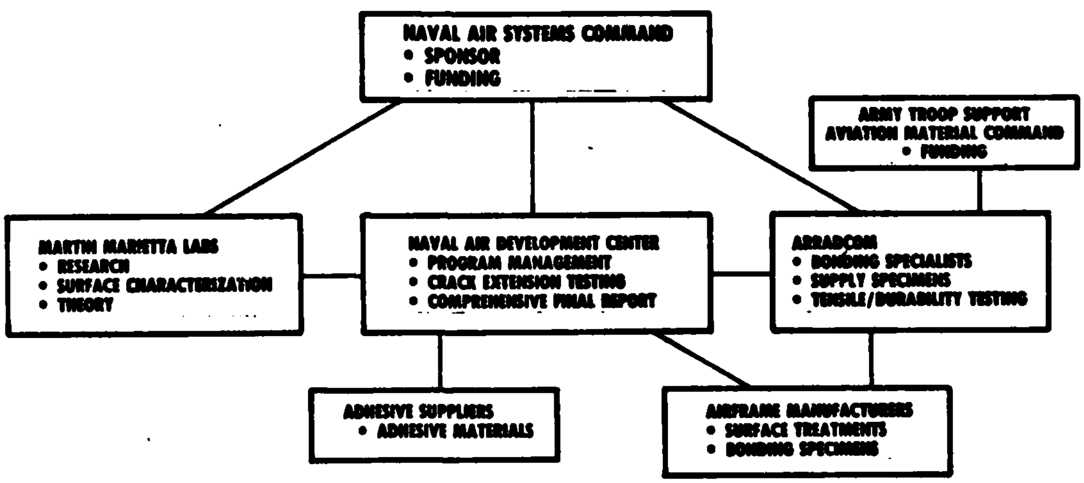

FIGURE 1. PARTICIPATING ORGANIZATIONS IN THE TITANIUM BONDING PRETREATMENT PROGRAM

A program that incorporates adhesive bond durability tests with surface characterization studies of titanium pretreatment processes is expected to resolve a number of problems and advance adhesive bonding technology.

TITANIUM PREBOND PROCESSES AND ADHESIVE SYSTEMS

The aircraft and space industry was surveyed to determine current production methods for pretreating titanium for adhesive bonding. Manufacturers representing the different pretreatment processes were invited to participate in the comprehensive program by pretreating and bonding specimen sets. Some recently developed processes were included in the eleven pretreatments and modifications studied in this program as follows:

ID		Temperature/Times
PF-1	Phosphate fluoride treatment after a $HF/HNO_3/NaSO_4$ pickle	R.T., 2 minutes
DP-2	Pasa-Jell 107M treatment after dry hone abrasion	R.T., 10-15 minutes
PF-3	Phosphate fluoride treatment after: (1) HNO_3 predip (2) $HF/HNO_3/NaSO_4$ pickle	R.T., 2 minutes
PF-4	Phosphate fluoride treatment after HF/HNO_3 pickle	R.T., 2 minutes
CA5-4	Chromic acid solution plus HF Anodize at 5 volts	R.T., 20 minutes
CA10-4	Chromic acid solution plus HF Anodize at 10 volts	R.T., 20 minutes
LP-6	Pasa-Jell 107 C7 treatment after: (1) liquid hone (2) alkaline clean	R.T., 15-20 minutes
DA-5	Dapco treat 4023/4000	R.T., 15 minutes
VA-7	VAST treatment	R.T., 15 seconds (maximum 15 minutes in solution)
TU-8	Turco 5578	190 F, 10 minutes
AP-9	Alkaline Peroxide treatment	130 F, 25 minutes

Complete bath preparation and process conditions contain proprietary information so only pretreatment types, solution temperatures and treatment times are included in this report.

The adhesive systems selected for pretreatment adhesive bond durability studies included two 250 F cure and two 350 F cure film materials as follows:

ID	Adhesive System	Cure Conditions
W1	FM300K/BR127	350 F, 40-50 p.s.i., 1 hour
W2	FM73M/BR127	250 F, 40 p.s.i., 1 hour
W3	M329/M329 Type II	350 F, 40-50 p.s.i., 1 hour
W4	EA9628H/BR127	250 F, 40 p.s.i., 1 hour

Adhesively bonded wedge specimen sets are identified by the prefix "W" followed by the number of the adhesive system. The same pretreatment and adhesive code systems are used in reports on the related studies by ARRADCOM and Martin Marietta.

WEDGE TEST EXPERIMENTAL PROCEDURE

All wedge specimen assemblies were prepared from 6 by 6 by 0.150-inch thick sheets of Ti-6Al-4V titanium supplied by ARRADCOM. Bonded assemblies were returned to NAVAIRDEVCEN for preparation and testing. The returned assemblies were cut into 1-inch wide specimens as shown in Figure 2.

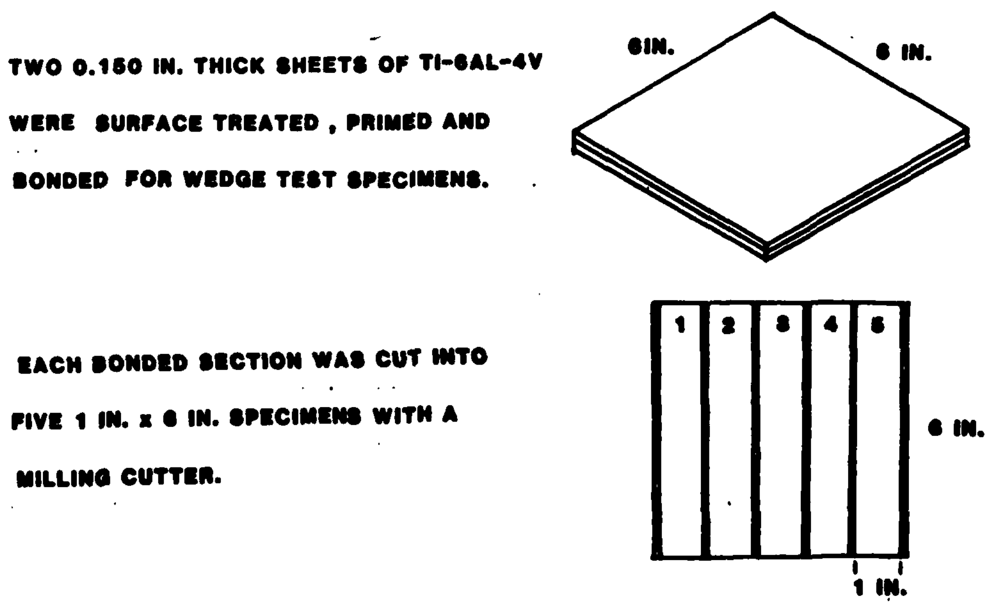

FIGURE 2. BONDING AND CUTTING STEPS IN THE PREPARATION OF TITANIUM WEDGE SPECIMENS

Conventional machining methods covered the adhesive bondlines with metal shavings making it difficult to determine crack tip locations during wedge testing. A wet grinding procedure was developed to remove metal from either side of the bondline leaving an 0.010-inch protrusion of the adhesive layer sandwiched between thin metal layers as shown in Figure 3. Dry belt sanding removed the protrusions and provided unobstructed bondlines for accurate crack tip readings.

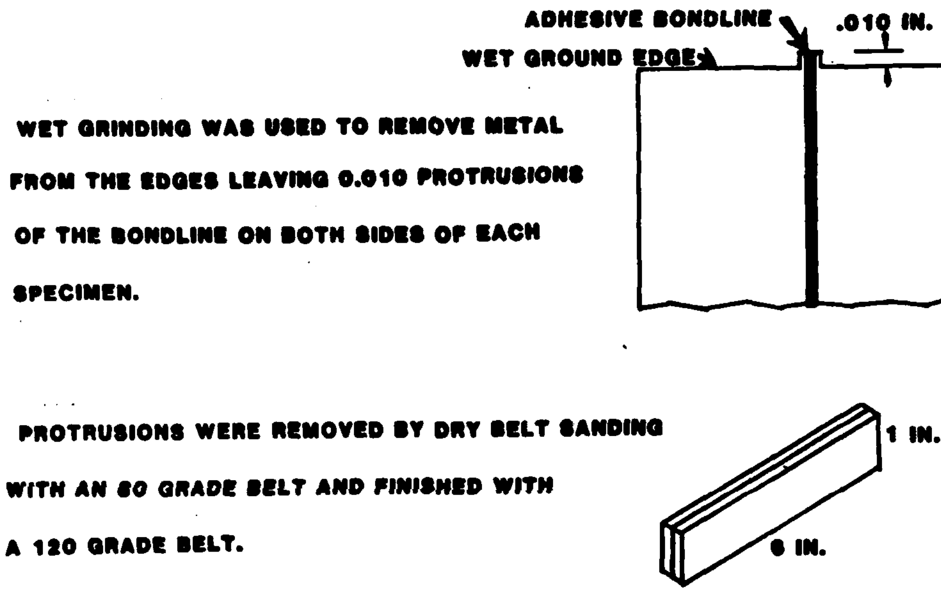

FIGURE 3. WET GRINDING AND DRY SANDING STEPS IN THE PREPARATION OF TITANIUM WEDGE SPECIMENS

Standard dimension 0.125-inch thick wedge specimens as described in ASTM Method D 3762 were used in this study. Wedges were fabricated from 303 stainless steel. One hour after inserting the wedges, initial crack lengths (a) were determined and marked on both sides of each specimen. Specimens were then exposed to the 140 F, 100% relative humidity (condensing) test conditions. Crack growth readings (Δa) were made after exposure times of 1, 4, 24, 48 and 96 hours, and 1, 2, 4 and 8 weeks. Crack tip locations were determined with a stereo binocular magnifier at 30X magnification. At completion of the 8-week crack growth test period specimens were split apart for failure mode analysis of bondline surfaces.

RESULTS AND DISCUSSION

Wedge crack extension measurements to evaluate adhesive bond durability are expressed either as crack growth during the exposure test period (Δa), or initial crack length plus growth (a +Δa). The crack growth method is satisfactory for pretreatment process control when only one pretreatment, one manufacturer and one adhesive system are involved. In a program involving several bonding facilities, however, bonding process variations, as well as surface pretreatment effects, contribute to wedge extension crack growth rates. Differences in initial crack openings for the same adhesive system are attributed to bonding process variations among the nine airframe manufacturers as well as surface pretreatment effects. Short initial crack openings that show higher strength bonded joints may be due to surface roughness, but do not reflect durability of prebond treatments of the titanium adherends. The shorter crack openings must withstand higher opening mode stresses at the crack tip than longer total crack openings regardless of growth during the exposure period. In addition, some pretreated surfaces started to deteriorate during the period between bonding and testing of specimens even though the test program was conducted to minimize the effect of premature failure. To offset these bonding variations, total crack length is also considered as an indication of pretreatment durability. Performance of the various surface pretreatments are discussed both in terms of conventional growth rates (a) and total crack openings (a +Δa). Wedge specimen bondline fracture surfaces are also discussed and illustrated. Strain energy release rate curves are given for selected wedge specimen sets.

CRACK GROWTH RATES

Crack extension growth rates (Δa) of prebond surface treatments with the 350 F cure FM300K/BR127 adhesive system (W1) exposed one hour to 140 F, 100% R.H. condition ranged from only 0.01 inch for the 10 volt chromic acid anodize with fluoride pretreatment (CA10-4) to 1.74 inches for the VAST pretreatment (VA-7). After 56 days exposure this range increased to 0.09 inch for CA10-4 specimens and 2.35 inches for VA-7 specimens. Curves of crack growth rates for the 56 day exposure test period separate into three groups as shown in Figure 4. The three phosphate fluoride pretreatments (PF-1, PF-3, and PF-4) along with the VAST surface preparation (VA-7) resulted in extremely high crack growth rates. DP-2 and DA-5 pretreated specimens had moderately low growth rates while extremely low growth rates were the result of CA10-4, CA5-4, TU-8, AP-9, and LP-6 pretreatments.

Pretreatment crack growth rates with the 250 F cure FM73M/BR127 adhesive system (W2) arbitrarily divide into two groups as shown in Figure 5. All PF-4 and VA-7 pretreated specimens failed within the first hour of exposure and the PF-3 specimens had an average crack growth of 1.59 inches after only one hour. There were no PF-1 pretreated specimens returned for evaluation with the FM73M/BR127 adhesive system. The other seven pretreatment results were closely grouped at the end of the 56 day exposure period. Crack growth ranged from 0.28 inches for the Dapcotreat 4000 pretreatment (DA-5) to 0.44 inches for both the chromic acid anodize pretreatments (CA5-4 and CA10-4).

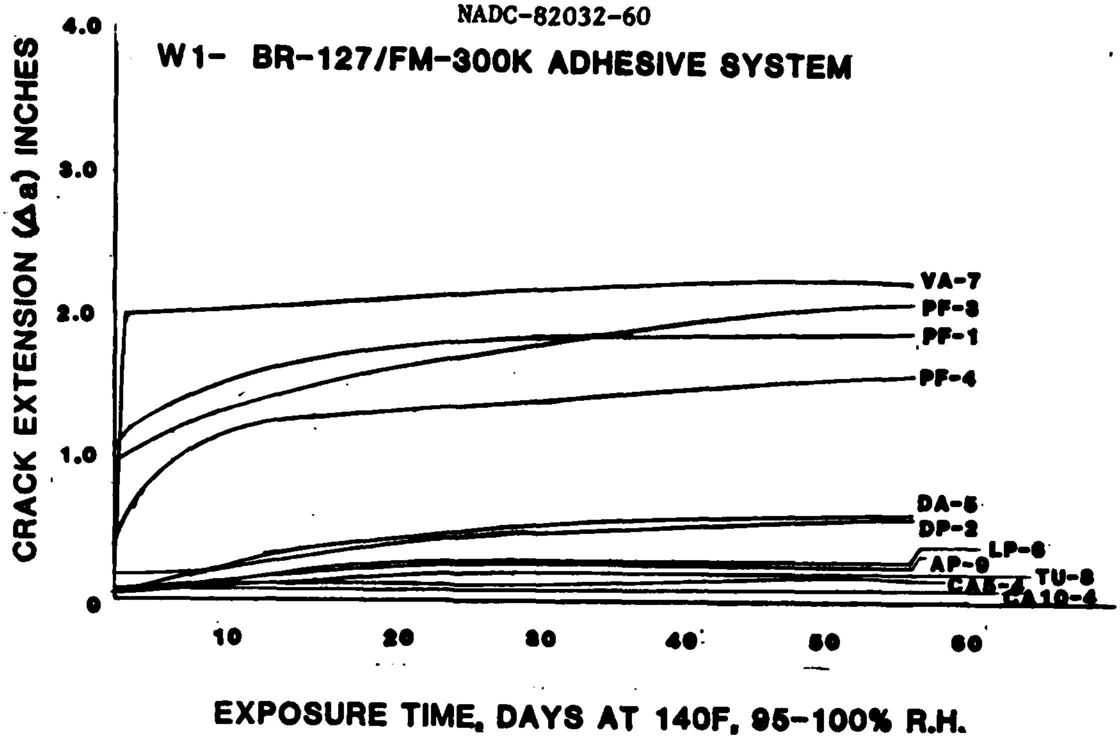

FIGURE 4. CRACK GROWTH RATES FOR TITANIUM BONDING PRETREATMENTS

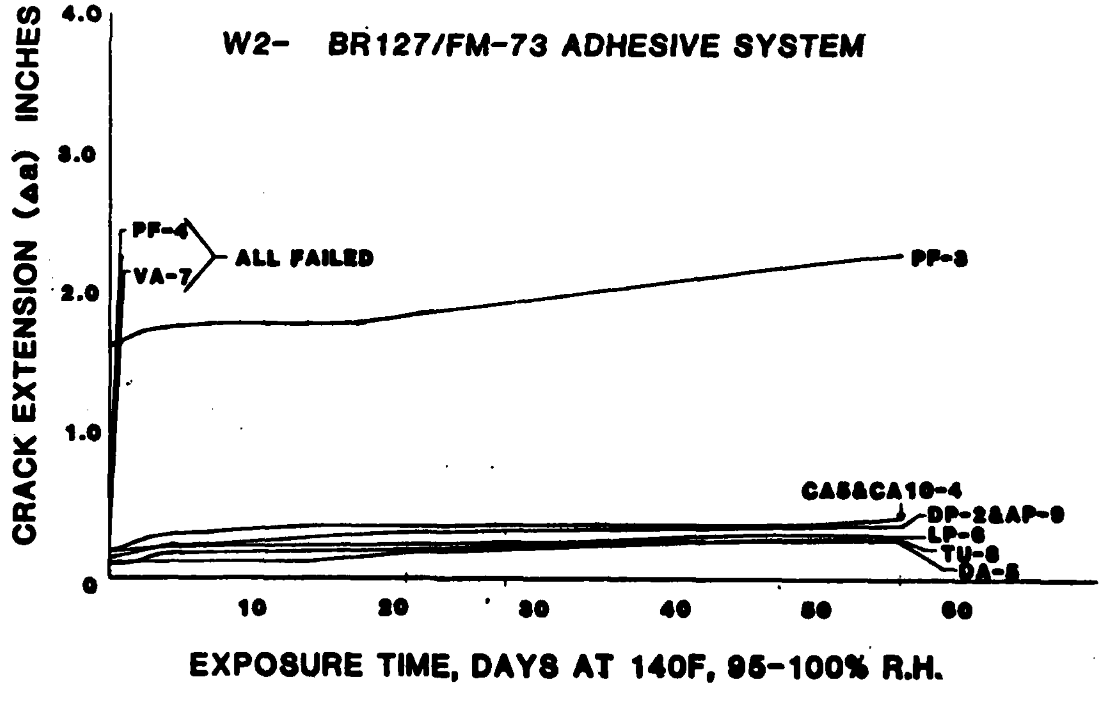

FIGURE 5. CRACK GROWTH RATES FOR TITANIUM BONDING PRETREATMENTS

Data for the pretreated specimens bonded with the other 350 F cure adhesive system, Metlbond 329/Metlbond 329 Type II Primer (W3) also separated into three groups after exposure testing. As shown in Figure 6 all PF-1 pretreated specimens failed within 14 days and the PF-4, PF-3 and VA-7 treated specimens had crack growth lengths of 1.73 to 1.81 inches after 56 days exposure.

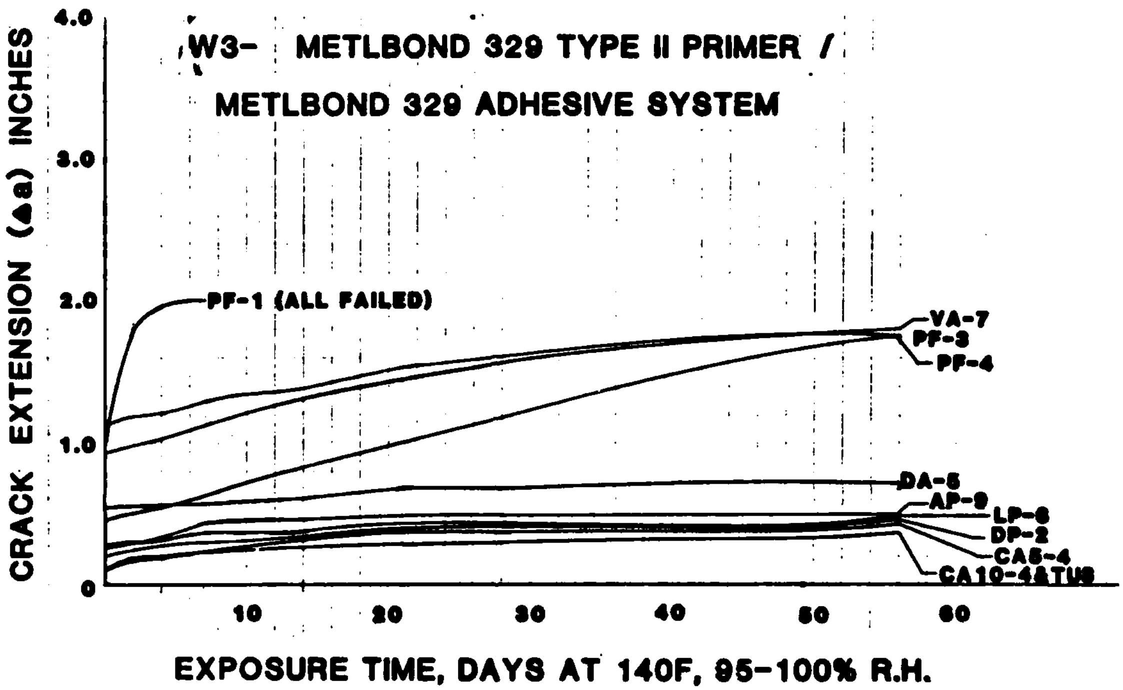

FIGURE 6. CRACK GROWTH RATES FOR TITANIUM BONDING PRETREATMENTS

Dapcotreat 4000 (DA-5) was the only pretreatment placed in this arbitrary middle group with an average crack growth of 0.73 inches after 56 days exposure. In the same test period the other six pretreatments had crack growths ranging from 0.35 inches for the 10 volt chromic acid anodize (CA10-4) to 0.49 inches for both the liquid hone Pasa Jell 107 (LP-6) and alkaline peroxide (AP-9) pretreatments.

Pretreatments bonded with the 250 F cure BR127/BA9628H adhesive system are divided into two groups based on crack growth test results as shown in Figure 7. All PF-1 pretreated specimens failed before or during the first hour of exposure testing. After 56 days PF-4 and VA-7 had respective crack growths of 2.73 and 2.81 inches. The moderately low crack growth pretreatments were DA-5, DP-2 and TU-8 with crack growths after 56 days of 0.59 to 0.63 inches. The remaining five pretreatments had low crack growth rates of 0.30 to 0.42 inches after 56 days exposure.

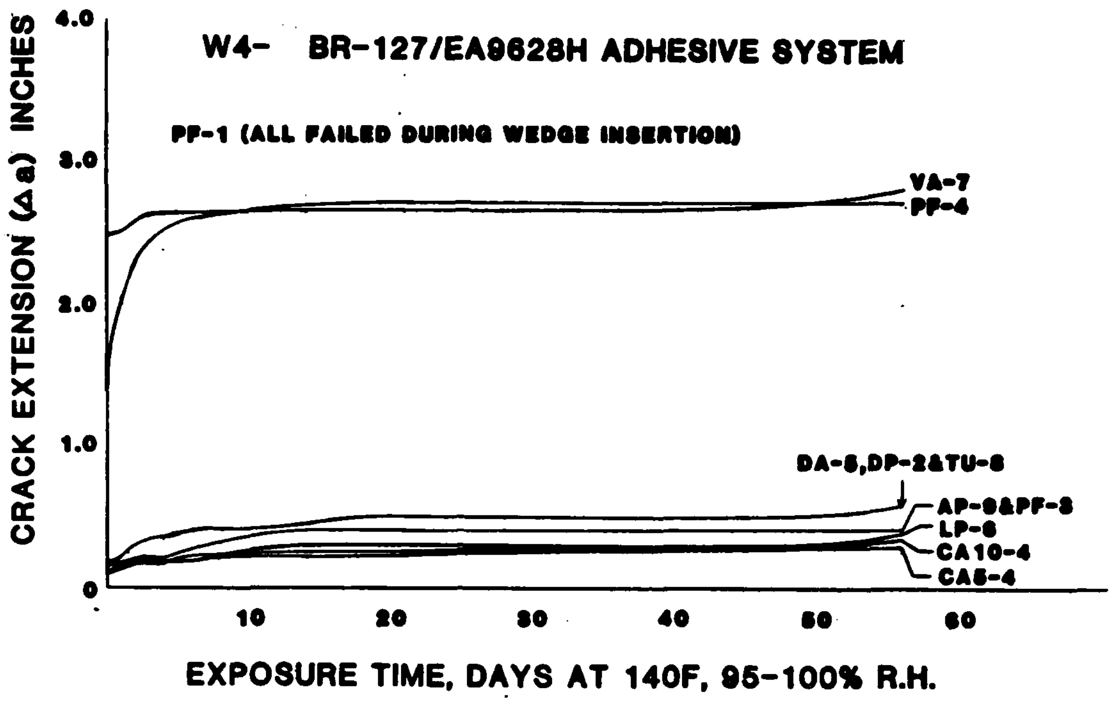

FIGURE 7. CRACK GROWTH RATES FOR TITANIUM BONDING PRETREATMENTS

The poor and marginal surface pretreatments show continued crack growth throughout the 56 day test period with the 350 F cure adhesives, FM300K and M329. Crack growth stopped, or was minimal, after 7-14 days exposure for most pretreatments with the 250 F cure adhesives, FM73M and EA9628H. Combining crack growth (Δa) data of the four adhesive systems gave the following durability rankings of the titanium pretreatments:

CA10-4	Most Durable
CA5-4	
TU-8	
LP-6	
AP-9	
DA-5	
DP-2	
PF-3	
PF-4	
VA-7	Least Durable
PF-1	

TOTAL CRACK OPENINGS

Total crack length measurements (a+Δa) of the eleven pretreatments with the 350 F cure adhesives after 24 hours exposure are shown in Figures 8 and 9.

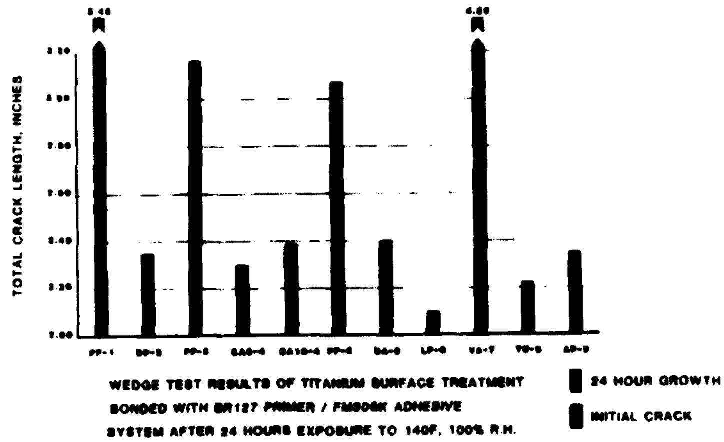

FIGURE 8. WEDGE TEST RESULTS OF BR127/FM300K AFTER 24 HOURS EXPOSURE to 140 F, 100% of R.H.

FIGURE 9. WEDGE TEST RESULTS OF METLBOND 329 TYPE II/329 AFTER 24 HOURS EXPOSURE TO 140 F, 100% R.H.

The liquid hone- Pasa Jell 107C pretreatment (LP-6) provided the lowest 24 hour crack opening with the FM300K adhesive, while the Turco 5578 (TU-8) method had the lowest total crack opening with the M329 adhesive system. Both of these pretreatments retained lowest average crack openings with the same adhesives after 56 days exposure. The LP-6 pretreatment also gave lowest total crack length results with the FM73M adhesive, while the chromic acid anodize pretreatment (CA5-4) was most effective with the EA9628H adhesive system. Total crack opening measurements (a+Δa) with all four adhesives after 56 days exposure testing are listed in Table I.

TABLE I. CRACK OPENING MEASUREMENTS

FM300K/BR127

Prebond Treatment ID	Initial Crack, in.	56 Day Crack Growth, in.	Total Crack Length, in. $\bar{x}$	STD. Dev. s
LP-6-W1	2.05	0.27	2.32 *	0.05
TU-8-W1	2.14	0.22	2.36 *	0.19
CA5-4-W1	2.24	0.17	2.41	0.08
CA10-4-W1	2.34	0.09	2.43	0.14
AP-9-W1	2.27	0.26	2.53	0.07
DP-2-W1	2.21	0.64	2.85	0.15
DA-5-W1	2.33	0.63	2.96	0.10
PF-4-W1	2.27	1.60	3.87	0.15
PF-1-W1	2.26	1.90	4.16	0.14
PF-3-W1	2.24	2.47	4.71	0.48
VA-7-W1	2.38	2.25	4.63	0.14

FM73M/BR127

Prebond Treatment ID	Initial Crack, in.	56 Day Crack Growth, in.	Total Crack Length, in. $\bar{x}$	STD. Dev. s
LP-6-W2	2.03	0.31	2.34 *	0.06
TU-8-W2	2.05	0.30	2.35 *	0.09
DA-5-W2	2.09	0.27	2.36 *	0.07
CA5-4-W2	1.94	0.44	2.38 *	0.05
CA10-4-W2	1.95	0.44	2.39 *	0.14
AP-9-W2	2.04	0.38	2.42 *	0.15
DP-2-W2	2.09	0.38	2.47	0.12
PF-3-W2	1.76	2.29	4.05	0.15
VA-7-W2	2.05	3.20	5.25 **	-
PF-4-W2	2.46	2.79	5.25 **	-
PF-1-W2	No specimens			

* No significant difference in average performance based on two-sided t-test at 95% confidence interval.

** Crack length extended entire length of specimens.

TABLE I. CRACK OPENING MEASUREMENTS (CONTINUED)

M329/M329 Type II

Prebond Treatment ID	Initial Crack, in.	56 Day Crack Growth, in.	Total Crack Length, in. x	STD. Dev. s
TU-8-W3	2.30	0.42	2.72 *	0.07
CA5-4-W3	2.32	0.43	2.75 *	0.18
CA10-4-W3	2.47	0.35	2.82	0.04
AP-9-W3	2.43	0.49	2.92	0.11
DA-5-W3	2.29	0.73	3.02	0.41
LP-6-W3	2.58	0.49	3.07	0.26
DP-2-W3	2.80	0.46	3.26	0.16
PF-4-W3	2.43	1.73	4.16	0.18
VA-7-W3	2.50	1.81	4.31	0.08
PF-3-W3	2.80	2.05	4.85	0.72
PF-1-W3	2.63	2.62	5.25 **	-

EA9628H/BR127

Prebond Treatment ID	Initial Crack, in.	56 Day Crack Growth, in.	Total Crack Length, in. x	STD. Dev. s
CA5-4-W4	1.90	0.30	2.20 *	0.02
LP-6-W4	1.87	0.38	2.25 *	0.06
CA10-4-W4	1.92	0.35	2.27	0.05
PF-3-W4	1.90	0.42	2.32	0.15
AP-9-W4	1.91	0.42	2.33	0.06
TU-8-W4	1.82	0.63	2.45	0.12
DP-2-W4	1.89	0.60	2.49	0.05
DA-5-W4	1.91	0.59	2.50	0.06
PF-4-W4	1.86	2.73	4.59	0.29
VA-7-W4	1.89	2.81	4.70	0.30
PF-1-W4	5.25 **	0.00	5.25 **	-

* No significant difference in average performance based on two-sided t-test at 95% confidence interval.

** Crack length extended entire length of specimens.

As noted in Table I there is no significant difference in average performance of the top two pretreatments with FM300K, M329 and EA9628H adhesives when applying the two-side t-test at a 95% confidence interval. The top five pretreatments with FM73M were the same based on this statistical analysis. However, there are significant differences between groups of effective and of poor pretreatments. Table II lists pretreatment averages of crack measurements calculated from combined results with all four adhesive systems.

TABLE II. COMBINED AVERAGES WITH ALL FOUR ADHESIVES

Ranking	Prebond Treatment ID	Average of 56 Day Total Crack Length, in. *
1	CA5-4	2.44
2	TU-8	2.47
3	CA10-4	2.48
4	LP-6	2.50
5	AP-9	2.55
6	DA-5	2.71
7	DP-2	2.77
8	PF-3	3.98
9	PF-4	4.47
10	VA-7	4.72
11	PF-1	4.89

* Combined average of specimens with all four adhesive systems.

Even though the total crack length method (a+Δa) is preferred, the only change in performance ranking from the crack growth method (Δa) was among the top three pretreatments.

All wedge test data collected in this program is listed by pretreatment in Tables III through XIII. Initial crack opening length (a), crack growth after each test period (Δa) and bondline thickness measurements are given for each specimen.

TABLE III. WEDGE TEST CRACK GROWTH DATA OF PF-1 PHOSPHATE FLUORIDE PA MODIFIED PRETREATMENT

Surface Treatment	Adhesive System	Specimen Identification Code		Bond Line Thickness, Inches	Initial Crack Length, Inches	Crack Growth Length (Inches) at Exposure Time: 1 Hr.	4 Hrs.	24 Hrs.	48 Hrs.	96 Hrs.	168 Hrs. (1 Week)	336 Hrs. (2 Weeks)	504 Hrs. (3 Weeks)	672 Hrs. (4 Weeks)	1344 Hrs. (8 Weeks)
Phosphate / Fluoride PF	FM300/BR127 W1	PF-1-W1	1A	.0042	2.07	.07	.29	.91	1.24	1.24	1.56	1.77	1.88	1.93	1.99
			1B	.0028	2.13	.08	.35	.96	1.14	1.39	1.42	1.65	1.83	1.97	1.97
			2A	.0044	2.27	1.41	1.54	1.56	1.56	1.46	1.53	1.63	1.70	1.76	1.76
			2B	.0037	2.19	1.41	1.54	1.55	1.55	1.60	1.70	1.78	1.93	2.00	2.00
			3A	.0025	2.33	1.08	1.08	1.08	1.08	1.17	1.21	1.59	1.59	1.75	1.86
			3B	.0032	2.37	1.32	1.41	1.41	1.41	1.44	1.51	1.64	1.64	1.64	1.84
			4A	.0035	2.48	.00	1.47	1.47	1.47	1.54	1.64	1.73	1.73	1.73	1.79
			4B	.0033	2.48	.00	1.35	1.33	1.35	1.33	1.59	1.59	1.59	1.59	1.59
			5A	.0035	2.15	.00	.29	.86	1.02	1.24	1.51	1.80	1.93	2.02	2.32
			5B	.0040	2.13	.06	.33	.78	.93	.93	1.58	1.73	1.88	2.04	2.14
		Average		.0035	2.26	.543	.965	1.193	1.275	1.346	1.512	1.691	1.770	1.849	1.900
	FM73/BR127 W2	PF-1-W2	1A, 1B, 2A, 2B, 3A, 3B, 4A, 4B, 5A, 5B	NO SPECIMENS RETURNED											
		Average													
	[illegible] W3	PF-1-W3	1A	.0056	2.43	.55	.63	1.43	1.63	1.78	1.88	TEST TERMINATED - CRACK EXTENDED ENTIRE LENGTH OF SPECIMEN			
			1B	.0064	2.40	.58	.69	1.47	1.61	1.65	1.77				
			2A	.0066	2.43	.72	.82	1.54	2.22	2.36	2.41				
			2B	.0072	2.47	.73	.83	1.51	2.19	2.26	2.33				
			3A	.0072	2.41	1.11	1.21	1.23	1.75	1.89	1.94				
			3B	.0070	2.41	1.14	1.14	1.28	1.68	1.83	1.97				
			4A	.0076	2.44	1.03	1.18	1.26	1.89	1.95	2.17				
			4B	.0061	2.45	.99	1.14	1.24	1.88	1.96	2.11				
			5A	.0068	2.93	1.68	1.68	1.68	1.68	1.71	1.76				
			5B	.0060	2.93	1.50	1.50	1.50	1.50	1.62	1.70				
		Average		.0067	2.63	1.00	1.08	1.40	1.80	1.90	2.00				
	[illegible]/BR127 W4	PF-1-W4	1A, 1B, 2A, 2B, 3A, 3B, 4A, 4B	Test Not Conducted On Specimens #1-4 Upon Insertion of Wedge - Specimens Split Into Two Halves											
			5A, 5B	Specimen #5 Split Into Two Halves After ½ Hr. Exposure In 100% R.H. Chamber @ [illegible]											
		Average													

TABLE IV. WEDGE TEST CRACK GROWTH DATA OF DP-2 DRY HONE PASA JELL 107C PRETREATMENT

Surface Treatment	Adhesive System	Specimen Identification Code		Bond Line Thickness, Inches	Initial Crack Length, Inches	CRACK GROWTH LENGTH (INCHES) AT EXPOSURE TIME									
						1 Hr.	4 Hrs.	24 Hrs.	48 Hrs.	96 Hrs.	168 Hrs. (1 Week)	336 Hrs. (2 Weeks)	504 Hrs. (3 Weeks)	672 Hrs. (4 Weeks)	1344 Hrs. (8 Weeks)
Dry Hone / Pasa Jell DP	FM300/BR127 W1	DP-2-W1	1 A	.0063	2.35	.05	.12	.19	.20	.20	.33	.53	.61	.70	.83
			B	.0039	2.39	.06	.14	.20	.27	.27	.34	.48	.62	.66	.82
			2 A	.0084	2.13	.09	.12	.12	.18	.18	.18	.32	.42	.50	.61
			B	.0073	2.23	.05	.14	.14	.17	.17	.21	.28	.40	.46	.66
			3 A	.0079	2.24	.09	.09	.13	.17	.17	.20	.37	.37	.52	.73
			B	.0092	2.05	.09	.09	.13	.17	.17	.23	.40	.53	.53	.74
			4 A	.0076	2.16	.12	.12	.21	.24	.24	.28	.28	.40	.50	.62
			B	.0068	2.16	.08	.08	.11	.19	.19	.21	.21	.35	.44	.59
			5 A	.0066	2.35	.06	.10	.10	.13	.19	.19	.25	.25	.45	.39
			B	.0037	2.39	.06	.10	.18	.23	.23	.26	.26	.26	.26	.38
		Average		.0068	2.21	.07	.11	.15	.19	.20	.24	.33	.42	.48	.64
	FM73/BR127 W2	DP-2-W2	1 A	.0049	2.10	.08	.16	.21	.21	.28	.28	.37	.44	.44	.55
			B	.0049	2.05	.08	.15	.21	.21	.28	.28	.37	.47	.47	.60
			2 A	.0109	2.11	.03	.07	.07	.11	.11	.17	.20	.20	.20	.20
			B	.0101	2.14	.05	.09	.09	.14	.14	.19	.36	.36	.36	.36
			3 A	.0131	2.08	.07	.11	.11	.17	.19	.19	.26	.26	.30	.30
			B	.0131	2.11	.06	.12	.12	.17	.20	.20	.27	.27	.33	.33
			4 A	.0106	2.14	.05	.07	.07	.14	.19	.19	.24	.24	.24	.24
			B	.0141	2.11	.06	.08	.08	.14	.21	.21	.26	.26	.26	.26
			5 A	.0096	2.03	.07	.12	.19	.24	.30	.30	.37	.42	.42	.48
			B	.0049	2.07	.06	.09	.13	.17	.33	.33	.39	.43	.43	.46
		Average		.0101	2.09	.06	.11	.13	.17	.23	.23	.29	.34	.35	.38
	Metlbond 329 329 Type II Primer W3	DP-2-W3	1 A	.0024	2.73	.10	.10	.13	.18	.21	.25	.32	.39	.39	.49
			B	.0036	2.66	.10	.16	.19	.22	.22	.26	.32	.41	.41	.46
			2 A	.0037	2.97	.07	.07	.10	.15	.15	.18	.24	.32	.36	.36
			B	.0045	2.98	.12	.12	.17	.17	.17	.21	.25	.29	.33	.38
			3 A	.0043	2.77	.40	.40	.50	.50	.50	.52	.60	.60	.60	.67
			B	.0043	2.83	.38	.38	.38	.38	.38	.48	.51	.57	.65	.68
			4 A	.0040	2.70	.05	.05	.09	.12	.12	.16	.21	.25	.39	.33
			B	.0040	2.82	.08	.08	.08	.11	.11	.11	.17	.22	.26	.26
			5 A	.0036	2.75	.36	.36	.36	.36	.36	.40	.45	.45	.49	.52
			B	.0030	2.76	.22	.22	.22	.26	.26	.33	.38	.38	.44	.47
		Average		.0037	2.80	.19	.20	.22	.26	.26	.29	.35	.40	.43	.46
	EA9628H/BR127 W4	DP-2-W4	1 A	.0043	1.86	.02	.05	.13	.32	.35	.39	.47	.47	.47	.67
			B	.0037	1.87	.05	.11	.19	.33	.35	.39	.49	.49	.49	.65
			2 A	.0049	1.87	.02	.08	.23	.43	.36	.36	.45	.45	.49	.59
			B	.0047	1.87	.08	.08	.19	.46	.32	.43	.49	.49	.54	.59
			3 A	.0047	1.87	.06	.06	.13	.29	.29	.36	.43	.43	.50	.57
			B	.0037	1.87	.06	.09	.16	.27	.30	.37	.42	.42	.49	.56
			4 A	.0049	1.93	.04	.08	.13	.23	.23	.34	.42	.42	.44	.55
			B	.0044	1.95	.04	.04	.12	.21	.21	.29	.37	.37	.37	.52
			5 A	.0037	1.90	.03	.03	.15	.29	.29	.40	.47	.47	.36	.69
			B	.0044	1.89	.03	.03	.17	.26	.29	.40	.45	.45	.45	.64
		Average		.0043	1.89	.041	.059	.162	.269	.287	.37	.45	.45	.48	.60

TABLE V. WEDGE TEST CRACK GROWTH DATA OF PF-3 PHOSPHATE FLUORIDE/HNO_3 (NITRIC ACID) PREDIP PRETREATMENT

Surface Treatment	Adhesive System	Specimen Identification Code	Bond Line Thickness, Inches	Initial Crack Length, Inches	CRACK GROWTH LENGTH (INCHES) AT EXPOSURE TIME									
					1 Hr.	4 Hrs.	24 Hrs.	48 Hrs.	96 Hrs.	168 Hrs. (1 Week)	336 Hrs. (2 Weeks)	504 Hrs. (3 Weeks)	672 Hrs. (4 Weeks)	1344 Hrs. (8 Weeks)
PHOSPHATE FLUORIDE PF	FM300/BR127 W1	PF-3-W1 1 A	.0050	2.22	1.06	1.26	1.34	1.39	1.39	1.53	1.79	1.94	2.10	TOTAL
		B	.0038	2.45	.73	.94	1.04	1.11	1.12	1.23	1.47	1.72	2.03	FAILURE
		2 A	.0034	2.17	.47	.50	.81	.98	1.10	1.25	1.45	1.56	1.69	1.94
		B	.0030	2.15	.43	.48	.90	1.04	1.16	1.28	1.43	1.57	1.62	1.94
		3 A	.0028	2.26	.24	.30	.73	.91	1.10	1.26	1.47	1.57	1.66	1.92
		B	.0027	2.37	.25	.30	.64	.91	1.07	1.26	1.40	1.55	1.69	1.87
		4 A	.0028	2.11	.47	.51	.68	.79	.90	1.09	1.28	1.48	1.71	2.18
		B	.0034	2.11	.38	.46	.64	.79	.98	1.14	1.36	1.51	1.66	2.19
		5 A	.0033	2.22	.53	.60	1.29	1.43	1.52	1.62	1.76	1.81	1.87	2.42
		B	.0030	2.32	.48	.54	1.27	1.38	1.47	1.57	1.67	1.75	1.81	2.31
		Average	.0033	2.24	.50	.60	.93	1.07	1.18	1.32	1.50	1.64	1.78	2.10
	FM73/BR127 W2	PF-3-W2 1 A	.0076	1.97	1.57	1.56	1.61	1.67	1.72	1.72	1.75	1.80	1.80	2.03
		B	.0074	1.71	1.60	1.69	1.81	1.81	1.81	1.86	1.92	1.97	1.97	2.21
		2 A	.0083	1.69	1.70	1.75	1.83	1.83	1.83	1.83	1.83	1.87	1.91	2.30
		B	.0074	1.72	1.61	1.68	1.77	1.77	1.79	1.83	1.83	1.87	1.93	2.28
		3 A	.0070	1.80	1.11	1.17	1.23	1.23	1.23	1.33	1.33	1.49	1.61	2.09
		B	.0064	1.81	1.12	1.20	1.31	1.31	1.35	1.39	1.39	1.56	1.81	2.02
		4 A	.0064	1.85	1.97	2.04	2.14	2.14	2.14	2.14	2.14	2.14	2.17	2.38
		B	.0074	1.68	1.91	1.98	2.13	2.13	2.13	2.13	2.13	2.16	2.22	2.46
		5 A	.0074	1.66	1.69	1.75	1.96	1.96	1.96	1.96	2.00	2.00	2.07	2.37
		B	.0064	1.77	1.67	1.77	1.77	1.77	1.77	1.77	1.79	1.79	1.95	2.25
		Average	.0072	1.76	1.59	1.66	1.76	1.76	1.77	1.80	1.81	1.87	1.95	2.29
	METLBOND 329 329 TYPE II Prime W3	PF-3-W3 1 A	.0070	2.58	.40	.83	.92	.98	1.02	1.10	1.19	1.36	1.49	1.73
		B	.0065	2.61	.46	.69	.75	.81	.81	.91	1.03	1.31	1.50	1.93
		2 A	.0068	3.23	.28	.75	1.14	1.20	1.23	1.28	1.44	1.54	1.66	TOTAL
		B	.0060	2.24	.28	.77	1.07	1.12	1.15	1.19	1.27	1.41	1.56	FAILURE
		3 A	.0065	3.28	.72	.72	.83	.91	.91	.94	1.07	1.15	1.19	1.24
		B	.0060	3.29	.71	.71	.76	.81	.85	.91	.98	1.05	1.11	1.22
		4 A	.0060	2.17	1.62	1.62	1.67	1.75	1.75	1.82	1.89	1.89	1.97	2.18
		B	.0062	2.16	1.62	1.62	1.78	1.83	1.83	1.83	1.87	1.89	1.98	2.17
		5 A	.0067	2.70	1.10	1.10	1.16	1.29	1.32	1.39	1.58	1.87	2.00	TOTAL
		B	.0066	2.79	1.03	1.03	1.08	1.17	1.21	1.33	1.49	1.81	2.01	FAILURE
		Average		2.80	.83	.99	1.16	1.18	1.20	1.28	1.38	1.53	1.65	1.75
	EA9628H/BR127 W4	PF-3-W4 1 A	.0036	1.90	.05	.11	.11	.11	.11	.20	.24	.24	[illegible]	.33
		B	.0065	1.88	.12	.16	.16	.16	.16	.22	.27	.27	.34	.40
		2 A	.0060	2.04	.07	.13	.13	.13	.16	.16	.19	.19	.19	.19
		B	.0062	1.96	.08	.14	.14	.14	.19	.22	.27	.27	.27	.27
		3 A	.0060	1.84	.04	.07	.07	.15	.20	.29	.34	.39	.39	.44
		B	.0064	1.84	.05	.09	.09	.09	.16	.25	.29	.39	.39	.43
		4 A	.0066	1.86	.06	.14	.14	.14	.18	.28	.40	.40	.40	.40
		B	.0060	1.90	.05	.11	.11	.11	.14	.24	.29	.29	.29	.35
		5 A	.0070	1.88	.05	.09	.54	.60	.64	.68	.73	.73	.73	.73
		B	.0055	1.88	.04	.08	.44	.60	.63	.68	.74	.74	.74	.74
		Average	.0062	1.90	.06	.14	.19	.22	.26	.32	.37	.39	.40	.42

TABLE VI. WEDGE TEST CRACK GROWTH DATA OF PF-4
PHOSPHATE FLUORIDE PRETREATMENT

Surface Treatment	Adhesive System	Specimen Identification Code		Bond Line Thickness, Inches	Initial Crack Length, Inches	CRACK GROWTH LENGTH (INCHES) AT EXPOSURE TIME									
						1 Hr.	4 Hrs.	24 Hrs.	48 Hrs.	96 Hrs.	168 Hrs. (1 Week)	336 Hrs. (2 Weeks)	504 Hrs. (3 Weeks)	672 Hrs. (4 Weeks)	1344 Hrs. (8 Weeks)
PHOSPHATE / FLUORIDE PF	FM300/BR127 W1	PF-4-W1	1 A	.0037	2.33	.07	.09	.27	.37	.68	.89	1.08	1.13	1.27	1.43
			B	.0050	2.27	.09	.11	.37	.55	.73	.95	1.21	1.39	1.55	1.71
			2 A	.0050	2.26	.72	.73	.91	.94	1.05	1.25	1.25	1.25	1.77	1.45
			B	.0032	2.31	.75	.89	.89	1.02	1.15	1.46	1.46	1.46	1.46	1.52
			3 A	.0053	2.28	.80	1.04	1.04	1.04	1.11	1.33	1.33	1.33	1.42	1.57
			B	.0058	2.34	.95	.95	.95	1.04	1.04	1.37	1.37	1.37	1.37	1.45
			4 A	.0042	2.25	.79	.90	.90	.96	.96	1.23	1.31	1.31	1.38	1.59
			B	.0058	2.24	.89	1.01	1.01	1.05	1.05	1.30	1.34	1.34	1.34	1.64
			5 A	.0029	2.27	.14	.14	.85	.89	1.02	1.21	1.41	1.59	1.66	1.93
			B	.0053	2.32	.10	.10	.83	.85	.96	1.09	1.23	1.47	1.47	1.66
		Average		.0046	2.27	.53	.59	.80	.87	.97	1.21	1.34	1.36	1.46	1.60
	FM73/BR127 W2	PF-4-W2	1 A	.0040	2.28										
			B	.0086	2.41	TEST TERMINATED AFTER 1 HOUR EXPOSURE TO									
			2 A	.0070	3.02										
			B	.0090	2.96	HUMIDITY CHAMBER - CRACK EXTENDED THE ENTIRE									
			3 A	.0090	2.31										
			B	.0084	2.25	LENGTH OF SPECIMENS AND SPLIT IN TWO HALVES									
			4 A	.0080	2.37										
			B	.0068	2.31										
			5 A	.0064	2.29										
			B	.0036	2.36										
		Average		.0068	2.46										
	METLBOND 329 / 329 TYPE II PRIMER W3	PF-4-W3	1 A	.0030	2.42	.55	.57	.67	.61	.68	.80	1.00	1.26	1.42	2.06
			B	.0037	2.49	.50	.50	.54	.54	.58	.64	.83	1.04	1.24	1.95
			2 A	.0040	2.51	.16	.32	.40	.44	.50	.55	.79	.92	1.01	1.68
			B	.0040	2.42	.23	.41	.47	.51	.55	.60	.80	.97	1.12	1.60
			3 A	.0044	2.49	.40	.42	.42	.48	.55	.67	.77	.91	1.07	1.52
			B	.0043	2.53	.36	.41	.41	.46	.51	.55	.69	.83	.87	1.41
			4 A	.0045	2.40	.61	.64	.64	.68	.72	.72	.85	1.00	1.08	1.63
			B	.0038	2.38	.63	.64	.64	.69	.72	.78	.83	.96	1.11	1.68
			5 A	.0042	2.35	.09	.12	.16	.25	.38	.54	.86	1.12	1.42	1.90
			B	.0025	2.30	.20	.23	.29	.36	.48	.58	.89	1.20	1.37	1.86
		Average		.0038	2.43	.37	.43	.46	.50	.57	.64	.83	.97	1.17	1.73
	EA9628H/BR127 W4	PF-4-W4	1 A	.0042	1.86	.09	.92	1.71	2.01	2.01	2.95	2.93	2.95	2.95	2.95
			B	.0038	1.85	.07	.68	1.65	1.84	1.96	3.11	3.11	3.11	3.11	3.11
			2 A	.0046	1.83	.07	1.75	2.30	2.30	2.30	2.38	2.41	2.48	2.48	2.48
			B	.0044	1.77	.07	1.86	2.33	2.33	2.33	2.44	2.49	2.49	2.49	2.49
			3 A	.0049	1.83	1.46	1.95	2.84	2.84	2.94	3.01	3.01	3.01	3.01	3.01
			B	.0047	1.85	1.21	2.14	2.78	2.83	2.83	2.99	2.99	2.99	2.99	2.99
			4 A	.0046	1.86	1.57	1.80	2.11	2.18	2.26	2.26	2.32	2.39	2.39	2.39
			B	.0040	1.87	1.49	1.74	2.07	2.18	2.25	2.25	2.30	2.45	2.45	2.45
			5 A	.0040	1.45	.76	1.21	2.09	2.22	2.33	2.41	2.86	2.86	2.86	2.86
			B	.0032	1.45	.70	1.21	2.09	2.35	2.48	2.48	2.48	2.57	2.57	2.57
		Average		.0038	1.76	.949	1.53	1.98	2.31	2.37	2.62	2.69	2.73	2.73	2.73

TABLE VII. WEDGE TEST CRACK GROWTH DATA OF CAS-4 CHROMIC ACID ANODIZE/FLUORIDE 5 VOLT PRETREATMENT

Surface Treatment	Adhesive System	Specimen Identification Code		Bond Line Thickness, Inches	Initial Crack Length, Inches	CRACK GROWTH LENGTH (INCHES) AT EXPOSURE TIME									
						1 Hr.	4 Hrs.	24 Hrs.	48 Hrs.	96 Hrs.	168 Hrs. (1 Week)	336 Hrs. (2 Weeks)	504 Hrs. (3 Weeks)	672 Hrs. (4 Weeks)	1344 Hrs. (8 Weeks)
Chromic Acid Anodize (5 Volt) CAS	FM300/BR127	CAS-4-W1	1A	.0026	2.29	.07	.07	.13	.13	.13	.13	.13	.13	.13	.22
	W1		1B	.0042	2.20	.08	.08	.20	.20	.20	.23	.23	.23	.23	.29
			2A	.0037	2.39	.05	.05	.05	.10	.10	.10	.10	.10	.10	.10
			2B	.0055	2.37	.00	.00	.00	.06	.06	.06	.06	.06	.06	.06
			3A	.0047	2.13	.04	.08	.08	.12	.12	.14	.14	.14	.14	.11
			3B	.0061	2.15	.05	.05	.05	.12	.12	.12	.12	.12	.12	.17
			4A	.0053	2.10	.06	.06	.06	.10	.10	.10	.15	.15	.15	.18
			4B	.0048	2.19	.03	.05	.05	.14	.14	.14	.14	.14	.14	.19
			5A	.0034	2.28	.00	.00	.12	.12	.12	.15	.15	.15	.15	.20
			5B	.0045	2.27	.00	.00	.00	.03	.03	.03	.08	.08	.08	.19
		Average		.0044	2.24	.04	.04	.07	.11	.11	.12	.13	.13	.13	.17
	FM73/BR127	CAS-4-W2	1A	.0036	1.97	.07	.14	.24	.29	.36	.36	.36	.36	.36	.42
	W2		1B	.0038	1.92	.14	.21	.29	.34	.42	.42	.42	.47	.47	.53
			2A	.0066	1.92	.08	.15	.25	.29	.29	.29	.29	.35	.35	.43
			2B	.0094	1.90	.09	.15	.24	.30	.30	.30	.30	.36	.36	.42
			3A	.0090	1.92	.10	.14	.22	.27	.27	.30	.30	.34	.34	.46
			3B	.0098	1.94	.12	.17	.23	.26	.26	.26	.26	.32	.32	.46
			4A	.0088	2.06	.08	.14	.21	.26	.26	.26	.26	.33	.33	.40
			4B	.0078	2.08	.09	.13	.20	.23	.23	.26	.26	.31	.31	.38
			5A	.0076	1.84	.13	.17	.24	.38	.28	.39	.39	.43	.43	.49
			5B	.0040	1.94	.09	.13	.21	.28	.30	.30	.30	.34	.34	.39
		Average		.0070	1.95	.09	.18	.21	.28	.29	.31	.31	.36	.36	.44
	Metlbond 329 / 329 Type II Primer	CAS-4-W3	1A	.0038	2.47	.12	.12	.20	.23	.27	.34	.39	.45	.47	.60
	W3		1B	.0034	2.45	.15	.21	.21	.28	.28	.39	.42	.47	.47	.53
			2A	.0032	2.33	.10	[illegible]	.20	.25	.27	.31	.38	.44	.46	.49
			2B	.0038	2.28	.13	[illegible]	.22	.24	.24	.30	.36	.44	.46	.52
			3A	.0040	2.35	.03	[illegible]	.10	.12	.14	.20	.25	.25	.28	.37
			3B	.0045	2.37	.00	[illegible]	.07	.08	.10	.16	.25	.31	.31	.37
			4A	.0044	2.21	.06	.09	.13	.15	.15	.18	.21	.24	.24	.30
			4B	.0044	2.23	.09	.13	.17	.17	.17	.20	.24	.31	.31	.35
			5A	.0045	2.25	.08	.01	.12	.14	.18	.18	.24	.32	.32	.32
			5B	.0030	2.24	.09	.09	.13	.18	.21	.25	.31	.36	.36	.44
		Average		.0039	2.32	.09	.1.	.15	.18	.20	.25	.30	.36	.37	.43
	EA9628H/BR127	CAS-4-W4	1A	.0048	1.91	.08	.08	.07	.13	.13	.27	.28	.27	.27	.27
	W4		1B	.0042	1.93	.03	.12	.10	.18	.18	.24	.29	.24	.24	.29
			2A	.0039	1.87	.10	.15	.20	.20	.22	.28	.28	.28	.33	.37
			2B	.0043	1.90	.07	.07	.11	.17	.17	.22	.22	.22	.27	.29
			3A	.0043	1.92	.07	.07	[illegible]	.17	.19	.24	.24	.24	.28	.28
			3B	.0048	1.91	.06	.14	[illegible]	.21	.21	.25	.25	.25	.29	.27
			4A	.0048	1.89	.07	.11	.17	.23	.23	.29	.29	.29	.31	.31
			4B	.0048	1.89	.08	.11	.17	.17	.17	.24	.24	.24	.29	.32
			5A	.0048	1.90	.09	.04	.09	.15	.15	.22	.22	.22	.22	.36
			5B	.0043	1.90	.06	.06	.09	.16	.16	.23	.23	.23	.25	.27
		Average		.0043	1.90	.062	.092	.126	.179	.183	.248	.248	.248	.275	.277

TABLE VIII. WEDGE TEST CRACK GROWTH DATA OF CA10-4 CHROMIC ACID ANODIZE/FLUORIDE 10 VOLT PRETREATMENT

Surface Treatment	Adhesive System	Specimen Identification Code	Bond Line Thickness, Inches	Initial Crack Length, Inches	CRACK GROWTH LENGTH (INCHES) AT EXPOSURE TIME 1 Hr.	4 Hrs.	24 Hrs.	48 Hrs.	96 Hrs.	168 Hrs. (1 Week)	336 Hrs. (2 Weeks)	504 Hrs. (3 Weeks)	672 Hrs. (4 Weeks)	1344 Hrs. (8 Weeks)
Chromic Acid Anodize (10 Volt) CA10	FM300/BR127 W1	CA10-4-W1 1 A	.0079	2.50	.00	.05	.05	.07	.07	.08	.08	.08	.08	.08
		B	.0032	2.52	.00	.05	.05	.07	.07	.07	.07	.07	.07	.07
		2 A	.0076	2.24	.00	.07	.07	.07	.07	.07	.10	.10	.10	.10
		B	.0079	2.33	.00	.03	.03	.03	.03	.03	.08	.08	.08	.08
		3 A	.0089	2.08	.00	.06	.06	.09	.09	.09	.09	.09	.09	.09
		B	.0084	2.15	.00	.08	.04	.11	.11	.11	.11	.11	.11	.11
		4 A	.0066	2.51	.00	.00	.03	.03	.03	.03	.03	.03	.03	.03
		B	.0066	2.47	.00	.00	.04	.04	.04	.04	.04	.04	.04	.04
		5 A	.0037	2.28	.06	.06	.11	.17	.17	.17	.17	.17	.17	.17
		B	.0050	2.29	.00	.00	.06	.14	.14	.17	.17	.17	.17	.17
		Average	.0065	2.33	.006	.03	.05	.08	.08	.08	.09	.09	.09	.09
	FM73/BR127 W2	CA10-4-W2 1 A	.0038	2.04	.08	.12	.12	.24	.29	.38	.38	.42	.42	.56
		B	.0064	2.05	.07	.10	.10	.29	.37	.37	.37	.44	.44	.48
		2 A	.0068	1.84	.06	.09	.14	.19	.21	.28	.28	.32	.32	.37
		B	.0082	1.88	.07	.11	.15	.21	.21	.26	.26	.29	.29	.36
		3 A	.0086	1.87	.10	.15	.22	.27	.27	.33	.33	.39	.39	.46
		B	.0084	1.86	.07	.10	.15	.25	.25	.25	.25	.31	.31	.37
		4 A	.0084	1.94	.10	.15	.19	.24	.29	.38	.38	.38	.38	.44
		B	.0066	1.96	.07	.13	.19	.25	.29	.34	.34	.34	.34	.44
		5 A	.0064	2.03	.10	.14	.20	.25	.30	.30	.30	.30	.30	.41
		B	.0042	2.07	.10	.14	.20	.27	.31	.33	.33	.33	.33	.47
		Average	.0068	1.95	.08	.12	.16	.24	.28	.32	.32	.35	.85	.44
	METLBOND 329 329 Type II Primer W3	CA10-4-W3 1 A	.0085	2.52	.11	.13	.13	.18	.22	.22	.23	.27	.30	.35
		B	.0098	2.50	.09	.08	.12	.17	.20	.24	.24	.27	.30	.34
		2 A	.0098	2.52	.02	.06	.10	.14	.14	.17	.23	.25	.25	.30
		B	.0104	2.50	.00	.00	.06	.12	.12	.15	.18	.23	.23	.28
		3 A	.0085	2.49	.02	.08	.11	.16	.16	.18	.21	.25	.25	.29
		B	.0101	2.42	.06	.09	.19	.17	.17	.20	.25	.30	.33	.36
		4 A	.0105	2.48	.08	.13	.13	.18	.18	.27	.27	.29	.29	.33
		B	.0096	2.42	.07	.17	.17	.23	.23	.27	.31	.33	.33	.38
		5 A	.0096	2.45	.11	.11	.17	.21	.26	.26	.29	.34	.34	.42
		B	.0085	2.40	.11	.11	.16	.22	.25	.32	.36	.36	.32	.46
		Average	.0098	2.47	.08	.10	.13	.18	.19	.23	.26	.29	.30	.35
	EA9628H/BR127 W4	CA10-4-W4 1 A	.0033	1.98	.03	.03	.03	.14	.19	.19	.19	.19	.19	.34
		B	.0036	1.91	.03	.03	.09	.17	.21	.21	.21	.21	.24	.38
		2 A	.0041	1.95	.02	.02	.10	.18	.18	.24	.24	.24	.24	.33
		B	.0040	2.01	.04	.04	.11	.19	.19	.25	.25	.25	.25	.33
		3 A	.0040	1.89	.04	.09	.15	.23	.23	.23	.23	.23	.25	.39
		B	.0042	1.91	.07	.10	.14	.21	.21	.21	.21	.24	.31	.38
		4 A	.0037	1.95	.04	.08	.14	.19	.19	.23	.23	.23	.29	.38
		B	.0037	1.96	.04	.04	.11	.18	.18	.24	.24	.24	.25	.33
		5 A	.0044	1.81	.05	.05	.10	.19	.24	.24	.24	.24	.26	.36
		B	.0032	1.85	.04	.02	.12	.16	.21	.24	.24	.24	.24	.38
		Average	.0038	1.92	.04	.056	.11	.18	.20	.23	.23	.24	.25	.35

TABLE IX. WEDGE TEST CRACK GROWTH DATA OF DA-5
DAPCOTREAT 4023/4000 PRETREATMENT

Surface Treatment	Adhesive System	Specimen Identification Code	Bond Line Thickness, Inches	Initial Crack Length, Inches	1 Hr.	4 Hrs.	24 Hrs.	48 Hrs.	96 Hrs.	168 Hrs. (1 Week)	336 Hrs. (2 Weeks)	504 Hrs. (3 Weeks)	672 Hrs. (4 Weeks)	1344 Hrs. (8 Weeks)
					CRACK GROWTH LENGTH (INCHES) AT EXPOSURE TIME									
Dapcotreat 4023/4000 DA	FM300/BR127 W1	DA-5-W1 1 A	.0041	2.29	.00	.00	.03	.14	.14	.30	.53	.64	.73	.75
		B	.0041	2.34	.03	.03	.07	.27	.27	.32	.46	.65	.73	.82
		2 A	.0036	2.19	.10	.10	.16	.23	.23	.34	.47	.47	.61	.75
		B	.0040	2.22	.06	.06	.15	.22	.22	.32	.44	.53	.61	.69
		3 A	.0032	2.42	.035	.035	.035	.12	.12	.12	.28	.35	.39	.53
		B	.0037	2.36	.06	.06	.09	.26	.26	.26	.33	.38	.45	.59
		4 A	.0033	2.43	.00	.00	.00	.07	.07	.18	.29	.37	.43	.36
		B	.0037	2.39	.07	.07	.07	.16	.16	.25	.37	.44	.48	.61
		5 A	.0041	2.27	.00	.00	.07	.07	.07	.07	.31	.31	.38	.52
		B	.0035	2.39	.00	.00	.03	.03	.03	.08	.24	.32	.35	.43
		Average	.0036	2.33	.036	.036	.070	.137	.137	.224	.372	.464	.518	.625
	FM73/BR127 W2	DA-5-W2 1 A	.0037	2.10	.03	.06	.06	.06	.09	.09	.09	.11	.18	.23
		B	.0071	2.11	.04	.04	.04	.04	.04	.04	.04	.10	.19	.24
		2 A	.0099	2.06	.05	.12	.12	.12	.14	.14	.14	.22	.24	.23
		B	.0099	2.12	.06	.10	.10	.10	.12	.12	.12	.20	.23	.29
		3 A	.0079	2.08	.04	.04	.09	.13	.13	.13	.13	.13	.19	.28
		B	.0117	2.07	.06	.06	.06	.15	.15	.15	.15	.20	.26	.33
		4 A	.0109	1.99	.03	.03	.03	.03	.09	.09	.09	.14	.19	.27
		B	.0084	2.01	.07	.07	.13	.13	.16	.16	.16	.21	.24	.27
		5 A	.0074	2.10	.08	.12	.17	.17	.21	.21	.21	.25	.25	.25
		B	.0082	2.20	.04	.04	.08	.13	.16	.16	.16	.23	.23	.29
		Average	.0080	2.08	.05	.07	.09	.11	.13	.13	.13	.18	.22	.27
	Metlbond 329 329 [illegible] W3	DA-5-W3 1 A	.0068	2.20	.04	.09	.16	.23	.23	.26	.30	.37	.42	.42
		B	.0072	2.25	.11	.11	.13	.17	.17	.17	.23	.34	.36	.42
		2 A	.0074	2.21	.42	.42	.44	.44	.44	.47	.50	.56	.56	.60
		B	.0078	2.28	.30	.30	.36	.36	.36	.40	.50	.45	.45	.57
		3 A	.0085	2.21	.52	.52	.56	.56	.59	.59	.67	.73	.73	.73
		B	.0090	2.19	.50	.54	.54	.54	.58	.58	.58	.65	.65	.75
		4 A	.0086	2.37	.35	.35	.38	.38	.38	.38	.43	.43	.43	.48
		B	.0088	2.35	.39	.39	.42	.42	.42	.42	.46	.51	.51	.61
		5 A	.0067	2.46	[illegible]	1.20	1.26	1.26	1.29	1.29	1.31	1.35	1.35	1.42
		B	.0073	2.40	[illegible]	1.05	1.09	1.09	1.16	1.16	1.16	1.22	1.22	1.27
		Average	.0078	2.30	.39	.50	.54	.55	.56	.57	.60	.66	.67	.73
	[illegible]/BR127 W4	DA-5-W4 1 A	.0044	1.98	.03	.03	.13	.24	.31	.36	.41	.41	.41	.52
		B	.0035	1.95	.06	.06	.17	.23	.30	.36	.41	.41	.41	.53
		2 A	.0033	1.92	.05	.09	.24	.31	.31	.42	.42	.42	.46	.53
		B	.0035	1.91	.07	.11	.27	.34	.34	.42	.49	.49	.52	.56
		3 A	.0032	1.92	.00	.06	.22	.27	.38	.39	.39	.43	.43	.53
		B	.0037	1.83	.07	.04	.23	.29	.35	.42	.48	.49	.49	.59
		4 A	.0037	1.90	.11	.18	.18	.29	.43	.49	.53	.56	.56	.63
		B	.0040	1.96	.06	.06	.15	.22	.41	.41	.47	.61	.61	.58
		5 A	.0037	1.89	.04	.04	.25	.34	.44	.50	.55	.60	.60	.72
		B	.0035	1.86	.06	.16	.31	.37	.43	.51	.65	.57	.57	.71
		Average	.0035	1.91	.055	.093	.217	.290	.363	.428	.46	.49	.50	.59

TABLE X. WEDGE TEST CRACK GROWTH DATA OF LP-6 PASA JELL 107C WET HONE PRETREATMENT

Surface Treatment	Adhesive System	Specimen Identification Code		Bond Line Thickness, Inches	Initial Crack Length, Inches	CRACK GROWTH LENGTH (INCHES) AT EXPOSURE TIME 1 Hr.	4 Hrs.	24 Hrs.	48 Hrs.	96 Hrs.	168 Hrs. (1 Week)	336 Hrs. (2 Weeks)	504 Hrs. (3 Weeks)	672 Hrs. (4 Weeks)	1344 Hrs. (8 Weeks)
Liquid Hone/ Pasa Jell LP	FM300/BR127 W1	LP-6-W1	1A	.0035	2.07	.05	.05	.13	.13	.13	.20	.20	.20	.20	.20
			B	.0073	2.08	.02	.02	.06	.06	.06	.19	.19	.19	.19	.19
			2A	.0073	1.98	.03	.05	.05	.12	.12	.12	.12	.29	.29	.29
			B	.0073	1.97	.00	.07	.07	.12	.12	.18	.18	.29	.29	.29
			3A	.0073	2.14	.00	.00	.00	.00	.00	.00	.18	.25	.25	.25
			B	.0073	2.14	.04	.04	.04	.04	.04	.04	.19	.25	.25	.25
			4A	.0068	2.03	.00	.05	.12	.12	.12	.17	.32	.32	.32	.32
			B	.0071	2.03	.00	.05	.05	.05	.05	.05	.27	.27	.27	.27
			5A	.0037	2.03	.00	.04	.08	.08	.08	.14	.32	.32	.32	.32
			B	.0073	2.02	.00	.03	.03	.03	.03	.07	.31	.32	.32	.32
		Average		.0065	2.05	.016	.04	.06	.08	.08	.12	.23	.27	.27	.27
	FM73/BR127 W2	LP-6-W2	1A	.0035	2.01	.07	.12	.12	.12	.16	.16	.20	.20	.20	.30
			B	.0079	1.93	.06	.11	.11	.14	.14	.14	.19	.23	.27	.38
			2A	.0091	2.06	.07	.11	.11	.11	.21	.21	.21	.21	.24	.38
			B	.0110	2.12	.03	.03	.03	.03	.09	.09	.09	.09	.23	.29
			3A	.0131	2.08	.03	.03	.07	.11	.16	.16	.16	.20	.22	.29
			B	.0130	2.07	.03	.03	.07	.07	.11	.11	.11	.11	.20	.27
			4A	.0094	1.98	.04	.04	.07	.07	.14	.14	.17	.21	.27	.29
			B	.0123	1.96	.05	.05	.10	.10	.14	.14	.18	.21	.25	.31
			5A	.0049	2.10	.06	.10	.14	.14	.19	.19	.23	.23	.23	.27
			B	.0091	2.00	.04	.04	.14	.14	.21	.21	.25	.28	.28	.33
		Average		.0094	2.03	.05	.07	.10	.10	.16	.16	.18	.20	.24	.31
	Metlbond 329 329 Type II Primer W3	LP-6-W3	1A	.0140	2.88	.42	.45	.45	.50	.52	.55	.55	.64	.64	.68
			B	.0079	2.22	.49	.52	.54	.54	.57	.61	.61	.68	.68	.69
			2A	.0193	2.47	.17	.24	.24	.24	.24	.32	.32	.39	.39	.49
			B	.0146	2.47	.14	.21	.21	.21	.21	.27	.27	.34	.34	.34
			3A	.0198	2.67	.07	.16	.16	.16	.16	.20	.20	.30	.30	.47
			B	.0192	2.75	.07	.07	.07	.07	.07	.13	.13	.18	.18	.23
			4A	.0161	3.12	.19	.26	.26	.26	.26	.26	.26	.33	.33	.44
			B	.0192	3.16	.16	.19	.19	.19	.19	.19	.19	.28	.28	.39
			5A	.0089	2.32	.32	.32	.32	.32	.50	.50	.50	.60	.60	.64
			B	.0146	2.33	.33	.39	.39	.39	.46	.46	.46	.52	.52	.60
		Average		.0155	2.58	.24	.28	.28	.29	.32	.35	.35	.43	.43	.49
	EA9628H/BR127 W4	LP-6-W4	1A	.0035	1.96	.05	.05	.15	.15	.20	.27	.31	.31	.31	.41
			B	.0089	1.89	.09	.09	.14	.14	.14	.23	.31	.31	.31	.42
			2A	.0074	1.88	.07	.07	.07	.16	.16	.16	.24	.31	.31	.37
			B	.0095	1.94	.05	.05	.05	.17	.17	.17	.23	.23	.23	.38
			3A	.0131	1.88	.04	.10	.10	.20	.20	.20	.27	.27	.27	.35
			B	.0130	1.87	.10	.16	.16	.24	.24	.24	.28	.28	.28	.34
			4A	.0094	1.82	.10	.10	.10	.20	.20	.25	.35	.35	.35	.39
			B	.0123	1.83	.08	.08	.08	.16	.16	.21	.28	.28	.28	.35
			5A	.0049	1.87	.03	.03	.07	.16	.16	.20	.29	.29	.29	.35
			B	.0091	1.79	.09	.09	.14	.22	.22	.27	.38	.38	.38	.34
		Average		.0094	1.87	.07	.09	.11	.18	.19	.22	.29	.30	.30	.38

TABLE XI. WEDGE TEST CRACK GROWTH DATA OF VA-7 VAST PRETREATMENT

Surface Treatment	Adhesive System	Specimen Identification Code		Bond Line Thickness, Inches	Initial Crack Length, Inches	CRACK GROWTH LENGTH (INCHES) AT EXPOSURE TIME: 1 Hr.	4 Hrs.	24 Hrs.	48 Hrs.	96 Hrs.	168 Hrs. (1 Week)	336 Hrs. (2 Weeks)	504 Hrs. (3 Weeks)	672 Hrs. (4 Weeks)	1344 Hrs. (8 Weeks)
VAST	FM300/BR127	VA-7-W1	1 A	.0044	2.38	1.23	1.71	1.71	1.75	1.83	1.88	1.88	1.97	1.97	2.17
VA	W1		B	.0070	2.37	1.39	1.71	1.71	1.71	1.72	1.78	1.78	1.89	1.89	2.06
			2 A	.0073	2.38	2.18	2.18	2.18	2.18	2.18	2.26	2.36	2.31	2.31	2.35
			B	.0090	2.39	2.16	2.16	2.16	2.16	2.19	2.23	2.23	2.28	2.28	2.28
			3 A	.0090	2.31	1.22	1.80	2.03	2.03	2.05	2.10	2.10	2.14	2.14	2.19
			B	.0095	2.26	1.39	1.71	2.03	2.07	2.13	2.17	2.17	2.21	2.21	2.21
			4 A	.0090	2.45	2.04	2.11	2.11	2.11	2.13	2.21	2.21	2.26	2.26	2.26
			B	.0077	2.51	2.07	2.13	2.13	2.13	2.17	2.23	2.23	2.27	2.27	2.27
			5 A	.0075	2.36	1.66	1.95	1.95	1.95	2.02	2.05	2.05	2.10	2.10	2.30
			B	.0038	2.41	1.79	2.11	2.11	2.11	2.16	2.20	2.20	2.29	2.29	2.41
		Average		.0074	2.38	1.74	1.96	2.01	2.02	2.06	2.11	2.11	2.17	2.17	2.25
	FM73/BR127	VA-7-W2	1 A	.0040	2.07	TEST TERMINATED AFTER 1HR. EXPOSURE TO HUMIDITY CHAMBER–									
	W2		B	.0086	2.11										
			2 A	.0090	2.04										
			B	.0110	1.95										
			3 A	.0110	2.10	CRACK EXTENDED THE ENTIRE LENGTH OF SPECIMENS AND SPLIT									
			B	.0110	2.10										
			4 A	.0104	2.03										
			B	.0090	2.11	IN TWO HALVES									
			5 A	.0084	1.98										
			B	.0044	2.06										
		Average		.0087	2.05										
	METLBOND 329 / 329 TYPE II PRIMER	VA-7-W3	A	.0065	2.51	.14	.48	.58	.65	.74	1.06	1.19	1.40	1.59	1.82
	W3		B	.0075	2.44	.21	.21	.48	.63	.75	.95	1.24	1.43	1.54	1.89
			A	.0080	2.45	.13	.13	.99	1.04	1.13	1.19	1.32	1.44	1.62	1.88
			B	.0090	2.39	.18	.23	1.03	1.11	1.19	1.28	1.43	1.59	1.72	1.95
			A	.0090	2.42	.14	.19	1.28	1.32	1.35	1.35	1.40	1.48	1.57	1.81
			B	.0080	2.44	.13	.13	1.24	1.28	1.31	1.31	1.35	1.49	1.64	1.74
			A	.0080	2.50	.09	.09	1.17	1.20	1.25	1.28	1.38	1.47	1.53	1.63
			B	.0070	2.53	.18	.23	1.16	1.23	1.25	1.29	1.39	1.43	1.53	1.76
			A	.0065	2.47	.19	.50	.67	.72	.80	.91	1.12	1.32	1.45	1.77
			B	.0055	2.58	.22	.34	.60	.67	.77	.94	1.17	1.37	1.49	1.85
		Average		.0075	2.50	.16	.26	.92	.99	1.05	1.16	1.30	1.44	1.57	1.81
	EA9628H/BR127	VA-7-W4	1 A	.0038	2.00	1.87	2.19	2.19	2.26	2.29	2.32	2.32	2.32	2.32	2.59
	W4		B	.0040	1.92	2.06	2.28	2.28	2.32	2.34	2.37	2.37	2.37	2.37	2.56
			2 A	.0040	1.84	2.14	FAILED	FAILED	FAILED	FAILED	FAILED	FAILED	FAILED	FAILED	FAILED
			B	.0043	1.90	2.37	FAILED	FAILED	FAILED	FAILED	FAILED	FAILED	FAILED	FAILED	FAILED
			3 A	.0044	1.92	2.04	2.18	2.18	2.24	2.24	2.24	2.24	2.24	2.24	2.29
			B	.0045	1.91	2.09	2.22	2.22	2.26	2.30	2.30	2.30	2.30	2.30	2.43
			4 A	.0044	1.81	1.35	2.69	2.75	2.78	2.82	2.87	2.87	2.87	2.87	3.04
			B	.0045	1.89	1.45	2.57	2.83	2.87	3.03	3.06	3.06	3.06	3.06	3.16
			5 A	.0044	1.89	2.74	2.87	2.87	2.91	3.06	3.06	3.06	3.06	3.06	3.25
			B	.0038	1.88	2.70	2.86	2.86	2.91	3.00	3.00	3.00	3.00	3.00	3.17
		Average		.0042	1.89	2.07	2.43	2.52	2.57	2.64	2.65	2.65	2.65	2.65	2.81

TABLE XII. WEDGE TEST CRACK GROWTH DATA OF TU-8
TURCO 5578 PRETREATMENT

Surface Treatment	Adhesive System	Specimen Identification Code	Bond Line Thickness, Inches	Initial Crack Length, Inches	CRACK GROWTH LENGTH (INCHES) AT EXPOSURE TIME 1 Hr.	4 Hrs.	24 Hrs.	48 Hrs.	96 Hrs.	168 Hrs. (1 Week)	336 Hrs. (2 Weeks)	504 Hrs. (3 Weeks)	672 Hrs. (4 Weeks)	1344 Hrs. (8 Weeks)
TURCO 5578 TU	FM300/BR127 W1	TU-8-W1 1 A	.0032	2.31	.04	.04	.09	.09	.09	.09	.09	.14	.17	.17
		B	.0050	2.24	.00	.00	.00	.00	.00	.00	.00	.00	.00	.00
		2 A	.0063	1.93	.00	.08	.14	.14	.18	.14	.14	.21	.26	.33
		B	.0066	1.95	.00	.08	.08	.08	.09	.14	.14	.16	.16	.20
		3 A	.0068	2.01	.07	.07	.13	.13	.13	.13	.13	.17	.17	.17
		B	.0073	2.05	.08	.08	.08	.14	.14	.16	.16	.16	.16	.16
		4 A	.0066	2.20	.03	.03	.07	.07	.07	.07	.07	.11	.11	.11
		B	.0063	2.24	.08	.08	.05	.05	.05	.05	.05	.13	.19	.19
		5 A	.0050	2.41	.06	.06	.16	.18	.18	.18	.18	.34	.34	.46
		B	.0037	2.22	.06	.06	.15	.20	.20	.24	.24	.30	.30	.46
		Average	.0057	2.14	.03	.05	.09	.10	.11	.12	.12	.17	.18	.22
	FM73/BR127 W2	TU-8-W2 1 A	.0035	2.01	.07	.12	.12	.29	.29	.24	.24	.24	.24	.24
		B	.0074	2.11	.10	.14	.14	.29	.29	.24	.24	.24	.24	.24
		2 A	.0099	2.22	.10	.10	.10	.10	.10	.16	.16	.16	.16	.20
		B	.0109	2.18	.05	.09	.09	.09	.09	.14	.14	.14	.14	.26
		3 A	.0119	1.98	.07	.12	.12	.12	.19	.19	.19	.19	.22	.22
		B	.0123	1.95	.09	.13	.13	.13	.16	.16	.16	.32	.32	.32
		4 A	.0116	1.90	.08	.25	.25	.25	.25	.25	.28	.28	.34	.52
		B	.0099	1.94	.08	.17	.17	.17	.20	.20	.28	.24	.24	.30
		5 A	.0037	2.12	.04	.04	.04	.04	.20	.24	.24	.24	.24	.29
		B	.0086	2.08	.13	.13	.13	.13	.24	.34	.34	.34	.34	.37
		Average	.0088	2.05	.08	.13	.13	.16	.21	.22	.22	.23	.25	.29
	METLBOND 329 329 TYPE II PRIMER W3	TU-8-W3 1 A	.0068	2.33	.13	.13	.18	.21	.21	.38	.44	.47	.47	.57
		B	.0098	2.28	.07	.07	.13	.22	.22	.31	.31	.36	.36	.46
		2 A	.0105	2.32	.08	.10	.14	.16	.16	.21	.21	.24	.24	.36
		B	.0110	2.29	.05	.05	.14	.16	.16	.20	.20	.25	.25	.48
		3 A	.0120	2.37	.11	.11	.11	.13	.13	.16	.16	.23	.23	.32
		B	.0120	2.35	.06	.06	.11	.13	.13	.20	.20	.24	.25	.32
		4 A	.0120	2.27	.18	.13	.13	.13	.13	.20	.24	.30	.35	.44
		B	.0110	2.27	.09	.09	.13	.18	.18	.18	.23	.33	.35	.44
		5 A	.0108	2.28	.11	.11	.14	.21	.21	.24	.24	.32	.37	.37
		B	.0076	2.29	.18	.16	.20	.28	.28	.29	.30	.35	.35	.42
		Average	.0103	2.30	.10	.10	.14	.17	.18	.24	.25	.31	.32	.42
	EA9628H/BR127 W4	TU-8-W4 1 A	.0054	1.76	.03	.07	.20	.24	.30	.36	.43	.43	.47	.59
		B	.0033	1.80	.10	.10	.17	.28	.34	.39	.47	.47	.50	.60
		2 A	.0049	1.78	.10	.10	.18	.67	.67	.73	.83	.83	.83	.83
		B	.0044	1.75	.12	.12	.18	.55	.60	.70	.78	.78	.78	.92
		3 A	.0049	1.87	.07	.07	.28	.43	.43	.52	.52	.52	.58	.65
		B	.0052	1.86	.04	.08	.22	.34	.34	.46	.46	.46	.58	.61
		4 A	.0047	1.79	.07	.09	.13	.21	.21	.34	.43	.49	.49	.63
		B	.0046	1.86	.07	.07	.12	.20	.20	.28	.36	.41	.41	.49
		5 A	.0042	1.89	.10	.10	.15	.20	.23	.30	.38	.38	.38	.49
		B	.0044	1.78	.06	.06	.12	.16	.18	.28	.34	.38	.38	.50
		Average	.0046	1.82	.076	.086	.175	.328	.35	.43	.50	.51	.54	.63

TABLE XIII. WEDGE TEST CRACK GROWTH DATA OF AP-9
ALKALINE PEROXIDE PRETREATMENT

Surface Treatment	Adhesive System	Specimen Identification Code		Bond Line Thickness, Inches	Initial Crack Length, Inches	CRACK GROWTH LENGTH (INCHES) AT EXPOSURE TIME									
						1 Hr.	4 Hrs.	24 Hrs.	48 Hrs.	96 Hrs.	168 Hrs. (1 Week)	336 Hrs. (2 Weeks)	504 Hrs. (3 Weeks)	672 Hrs. (4 Weeks)	1344 Hrs. (8 Weeks)
ALKALINE PEROXIDE AP	FM300/BR127 W1	AP-9-W1	1A	.0026	2.37	.03	.03	.06	.09	.12	.12	.14	.14	.20	.20
			B	.0040	2.27	.04	.04	.09	.13	.24	.24	.24	.24	.31	.31
			2A	.0043	2.20	.04	.04	.08	.11	.18	.11	.21	.30	.30	.30
			B	.0048	2.16	.05	.05	.11	.16	.16	.16	.20	.20	.20	.20
			3A	.0050	2.40	.04	.04	.04	.10	.10	.10	.16	.16	.16	.16
			B	.0050	2.42	.04	.04	.04	.08	.08	.08	.12	.12	.12	.12
			4A	.0050	2.16	.06	.07	.07	.14	.14	.14	.21	.27	.32	.32
			B	.0044	2.20	.04	.04	.11	.17	.22	.22	.26	.32	.38	.38
			5A	.0047	2.21	.06	.06	.16	.22	.22	.22	.27	.34	.34	.34
			B	.0028	2.34	.04	.04	.09	.14	.14	.14	.21	.24	.25	.25
		Average		.0042	2.27	.04	.04	.08	.13	.15	.15	.20	.23	.26	.26
	FM73/BR127 W2	AP-9-W2	1A	.0080	2.21	.05	.10	.16	.16	.17	.24	.32	.32	.32	.32
			B	.0070	2.16	.06	.12	.18	.18	.18	.22	.25	.25	.25	.25
			2A	.0070	2.11	.05	.09	.09	.14	.14	.19	.24	.24	.24	.24
			B	.0084	2.06	.07	.13	.12	.17	.17	.24	.32	.32	.32	.32
			3A	.0080	1.93	.03	.09	.15	.15	.15	.23	.29	.29	.29	.29
			B	.0088	1.90	.06	.12	.13	.12	.12	.20	.28	.56	.56	.56
			4A	.0092	2.08	.05	.13	.29	.29	.29	.29	.40	.63	.63	.63
			B	.0070	2.03	.04	.11	.16	.16	.16	.21	.29	.46	.46	.46
			5A	.0070	1.96	.06	.12	.19	.27	.27	.33	.33	.44	.44	.44
			B	.0036	1.96	.00	.06	.16	.22	.22	.28	.28	.35	.35	.35
		Average		.0070	2.04	.05	.11	.16	.19	.19	.24	.30	.38	.38	.38
	METLBOND 329 329 TYPE II PRIM W3	AP-9-W3	1A	.0055	2.44	.12	.19	.23	.29	.29	.42	.42	.42	.42	.42
			B	.0090	2.38	.19	.25	.28	.35	.41	.52	.52	.52	.52	.52
			2A	.0056	2.47	.17	.26	.29	.35	.38	.48	.48	.61	.69	.69
			B	.0065	2.43	.18	.26	.30	.36	.42	.42	.49	.57	.57	.57
			3A	.0058	2.43	.23	.29	.31	.38	.38	.45	.48	.48	.48	.48
			B	.0056	2.47	.19	.24	.28	.34	.37	.43	.48	.48	.48	.48
			4A	.0060	2.47	.12	.16	.21	.28	.28	.35	.38	.43	.43	.43
			B	.0056	2.49	.14	.18	.23	.29	.33	.42	.46	.46	.46	.46
			5A	.0060	2.35	.13	.19	.22	.29	.29	.32	.41	.41	.41	.41
			B	.0050	2.37	.17	.21	.27	.35	.38	.46	.48	.48	.48	.48
		Average		.0061	2.43	.16	.22	.26	.33	.35	.43	.46	.49	.49	.49
	EA9628H/BR127 W4	AP-9-W4	1A	.0030	1.89	.04	.09	.17	.23	.29	.34	.46	.46	.46	.46
			B	.0036	1.93	.08	.13	.13	.16	.16	.21	.34	.43	.43	.43
			2A	.0040	1.88	.04	.12	.18	.23	.27	.31	.37	.37	.37	.37
			B	.0040	1.95	.04	.08	.11	.17	.21	.27	.39	.39	.39	.39
			3A	.0040	1.93	.08	.13	.13	.19	.21	.32	.41	.41	.41	.41
			B	.0040	1.96	.11	.17	.17	.23	.23	.30	.52	.52	.52	.52
			4A	.0036	1.88	.04	.08	.16	.19	.19	.32	.47	.47	.47	.47
			B	.0028	1.85	.05	.13	.22	.25	.25	.39	.46	.46	.46	.46
			5A	.0040	1.90	.06	.12	.19	.24	.29	.33	.42	.42	.42	.42
			B	.0034	1.96	.00	.07	.14	.18	.23	.28	.36	.36	.36	.36
		Average		.0036	1.91	.05	.11	.16	.20	.23	.30	.42	.42	.42	.42

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WEDGE TEST FRACTURE MECHANICS

Crack growth data is more useful for comparison to other studies when converted to strain energy release rate values. The strain energy release rate G_I (in.-lbs./in.^{2}), also called the crack extension force, is the apparent force at the wedge specimen crack tip due to an opening mode stress. Basic work by Ripling and Mostovoy (references (d) and (e)) led to an equation for G_I calculations for bonded uniform double cantilever beam (DCB) specimens. The resultant strain energy release rate formula with parameter values for wedge specimens used in this study is as follows:

$$G_I = \frac{y^2 M h^3 \quad (3\ (a+0.6h)^2 + h^2)}{16\ ((a + 0.6h)^3 + ah^2)\ ^2}$$

Where:

G_I = Strain Energy Release Rate, in.-lb./in.^{2}

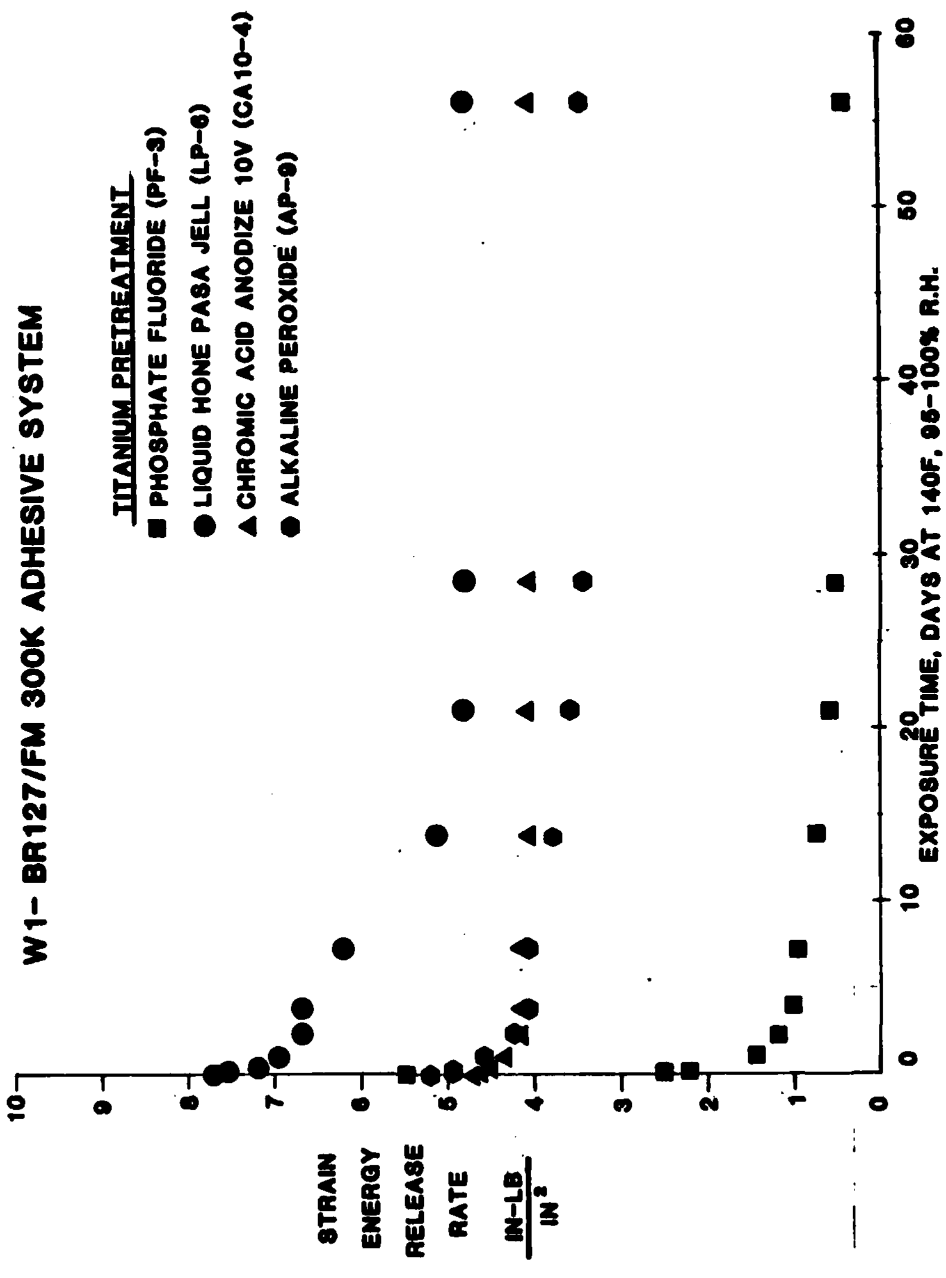
y = Displacement at Load Point, Inches = 0.125 in.

a = Distance from Load Point to Crack Tip, Inches

h = Height of One Beam, Inches = 0.150 in.

M = Modulus of Adherend, lb./in.^{2} = 16,500,000 lb./in.^{2}

Efforts by Marceau and others (references (f) to (h)) resulted in the development and application of the thin adherend DCB specimens, or wedge test specimens, to control adherend surface quality for adhesive bonding. The wedge test method is good for qualitative determinations, but not used for quantitative studies because of plastic deformation of thin metal adherends. However, the strain energy release rate calculation is an effective method to show apparent force at the crack tip. Figures 10 and 11 are plots of uncorrected G_I values as a function of exposure time. No attempt was made to adjust strain energy release rate values of the 0.150-inch thick titanium adherends due to the complexity of plastic deformation rates. Calculated values are higher than actual strain energy release rates with an increasing differential between calculated and actual rates at longer exposure times. It is apparent from the above plots that liquid hone Pasa Jell, 10 volt chromic acid anodize and alkaline peroxide pre-treatments have much higher strain energy release rates (calculated or actual) than the PF-1 phosphate fluoride pretreated with both FM300K and FM73M adhesive systems.



STRAIN ENERGY RELEASE RATE AS A FUNCTION OF EXPOSURE TIME FOR TITANIUM BONDING PRETREATMENTS

FIGURE 10

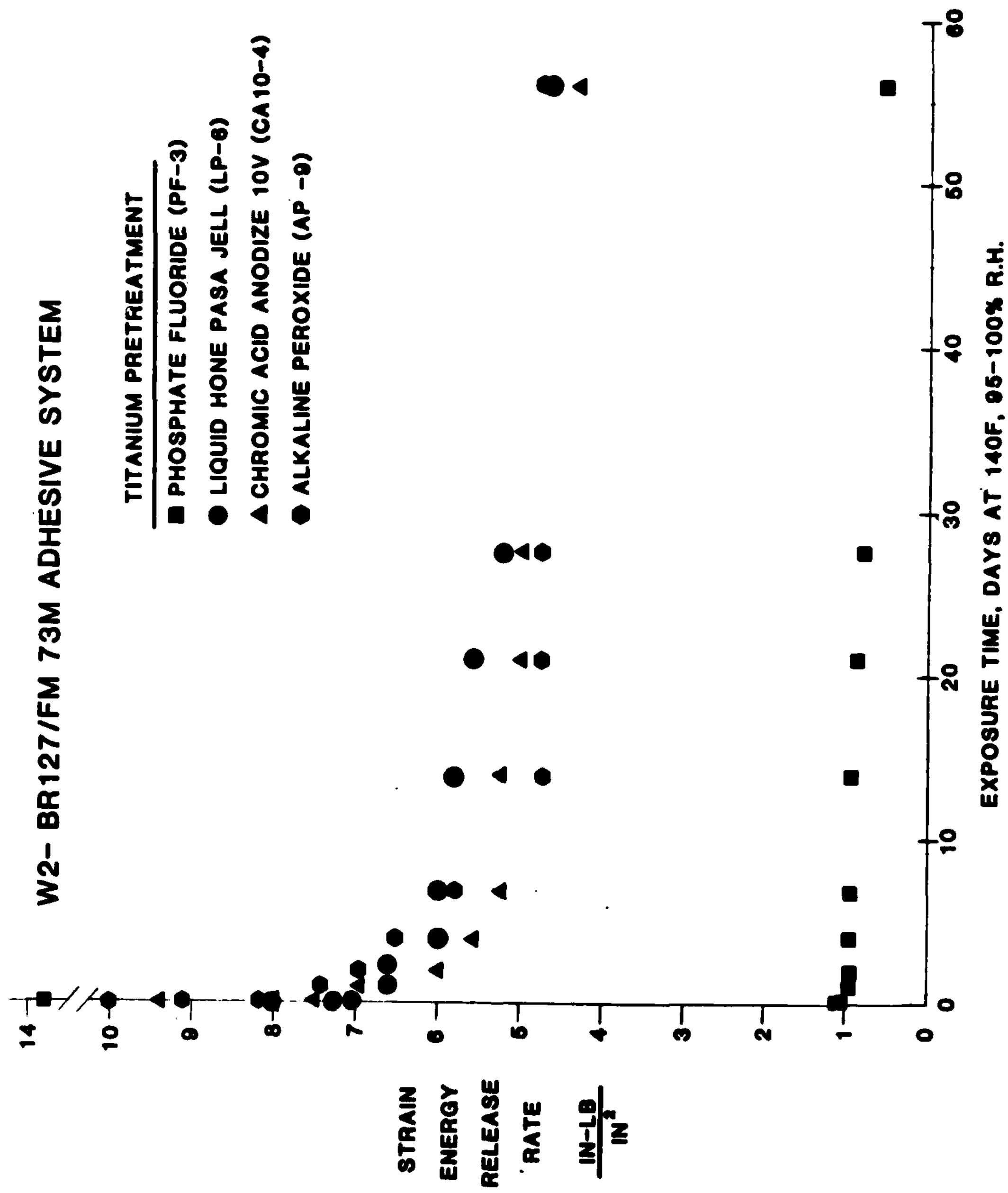
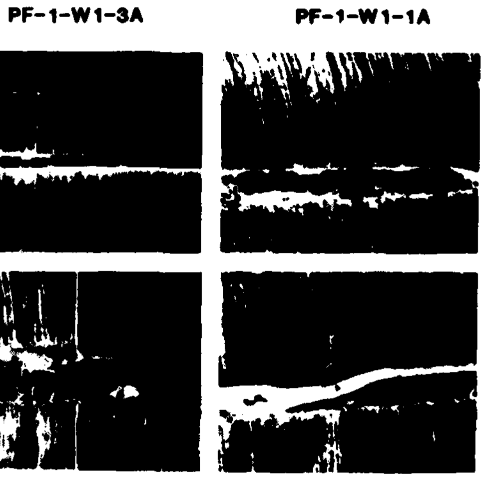


FIGURE 11

STRAIN ENERGY RELEASE RATE AS A FUNCTION OF
EXPOSURE TIME FOR TITANIUM BONDING PRETREATMENTS

FRACTURE SURFACES

The initial fractures that result from driving the wedges into the specimens are cohesive failures through the adhesive layer as shown in Figures 12 and 13. When exposed to 140 F, 100% R.H. conditions, the cracks transition into adhesive debonding as shown with the phosphate fluoride pretreatment (PF-1) in Figure 12.



TITANIUM WEDGE SPECIMENS WITH AS MACHINED EDGES
BR127 PRIMER/FM300K ADHESIVE BONDLINE
AFTER 24 HOURS EXPOSURE TO 140F, 100% R.H.

FIGURE 12. TITANIUM WEDGE SPECIMENS WITH AS MACHINED EDGES

The 7.5X and 32X magnification views of specimen PF-1-W1-3A both show the cohesive failure mode caused by wedge insertion. This cohesive failure zone is apparent from the left edge of the photos to the vertical scribe mark. Cohesive failure is shown in the 20X magnification of specimen PF-1-W1-1A, but transitions into a classic adhesive failure debond in the 30X magnification view at the initial crack tip mark. Figures 13 and 14 show initial cohesive fracture followed by adhesive failure growth with other pretreatments and adhesive systems. The wet ground-dry belt sand finish method as shown in Figure 3 can be observed on specimen edges in Figures 13 and 14. The wet ground-dry sand preparation technique provides distinct adhesive/metal interfaces on specimen edges and simplifies crack tip determinations since adhesive failures followed along these interfaces.

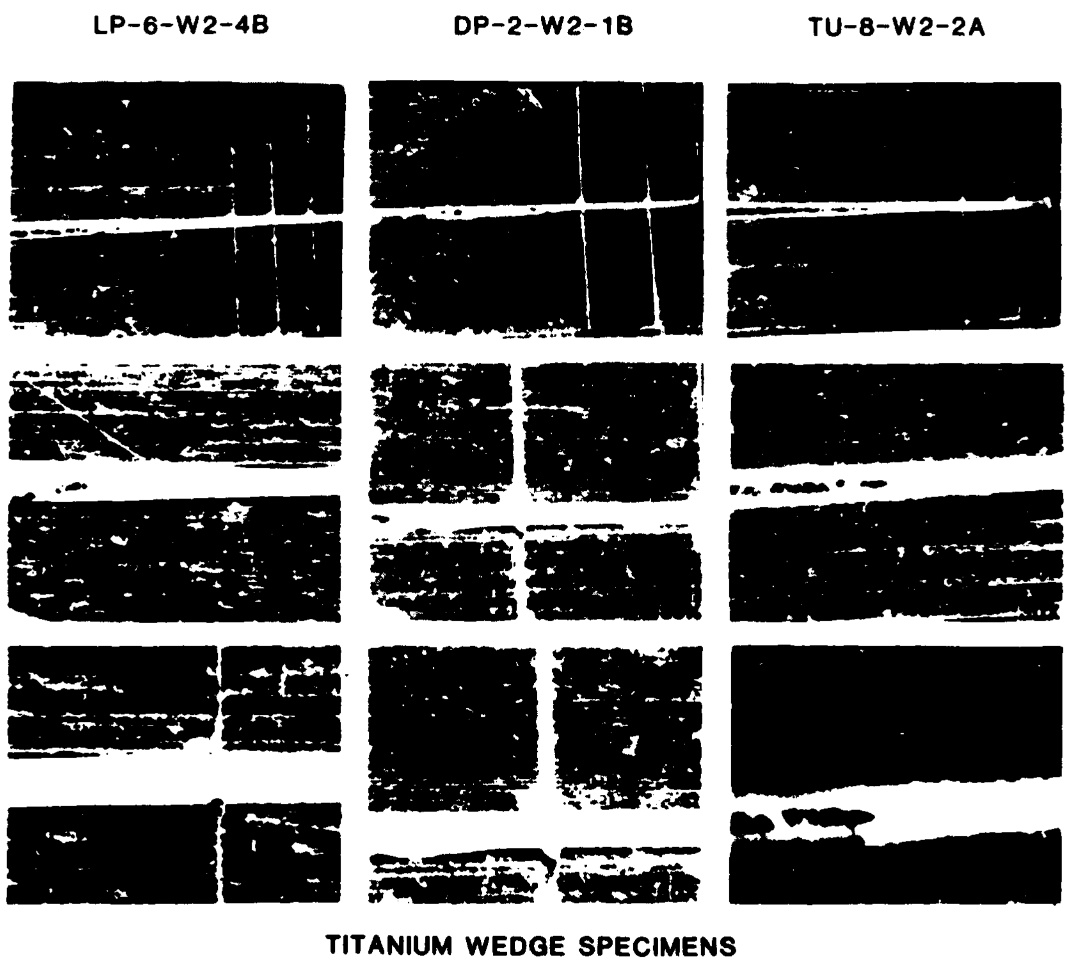


FIGURE 13. WEDGE SPECIMENS OF BR127/FM73 AFTER 24 HOURS EXPOSURE TO 140 F, 100% R.H.

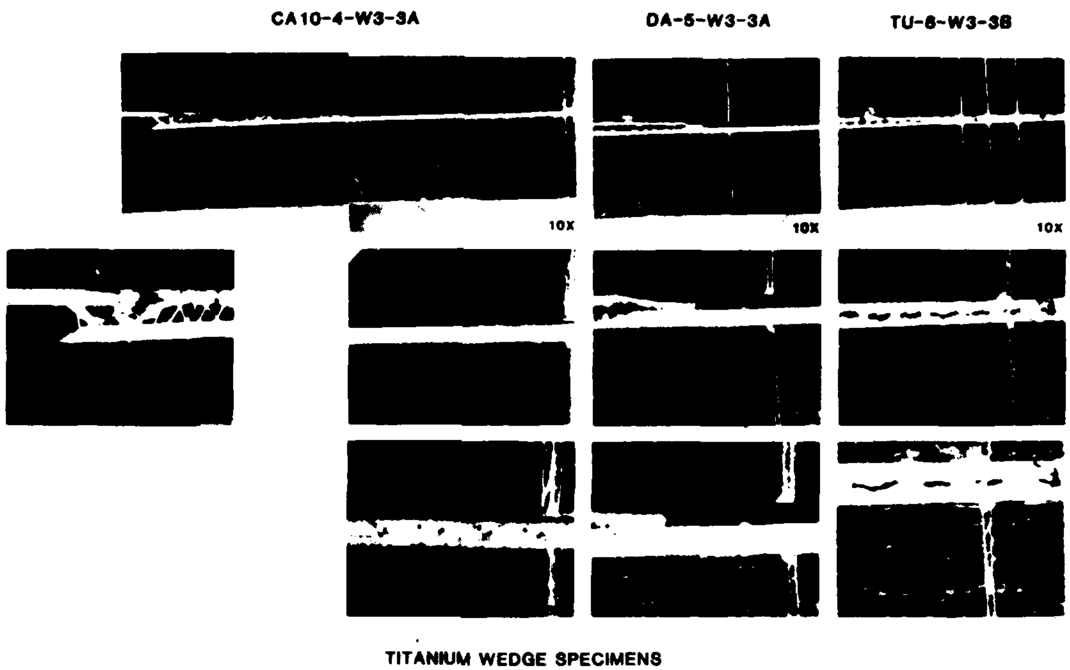


FIGURE 14. WEDGE SPECIMENS OF METLBOND 329 TYPE II/329 AFTER 24 HOURS·EXPOSURE TO 140 F, 100% R.H.

Initial cracks plus growth after 96 hours test exposure are shown in the montages of Figure 15 and illustrate crack growth rates for PF, DA and LP pretreatment processes.

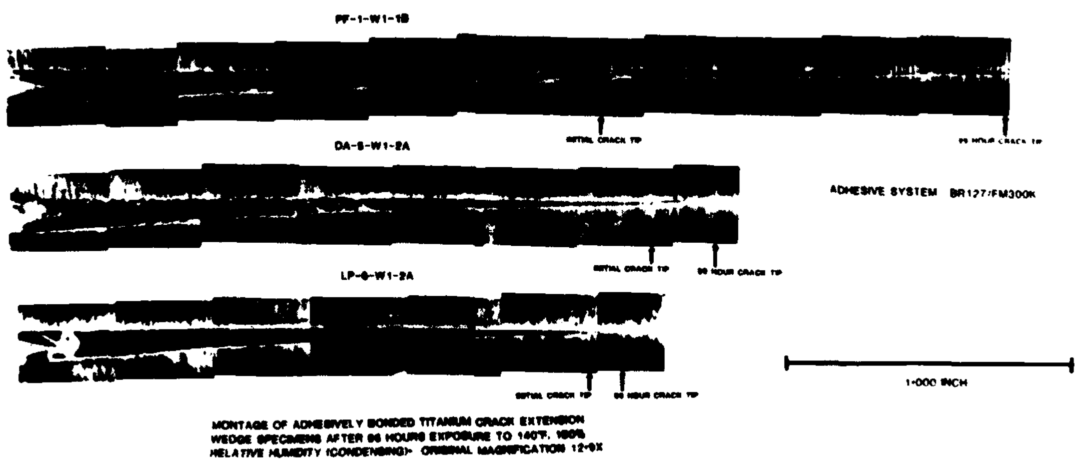


FIGURE 15. MONTAGE OF CRACK EXTENSION IN WEDGE SPECIMEN

Specimens were split open after completion of the 56 day exposure period for failure mode analysis. Figures 16 through 26 are photographs of opened wedge specimen sets of all eleven pretreatment processes bonded with the BR127/FM300K adhesive system. Observe that wedge crack growths during exposure are mostly adhesive failure as shown in the center sections of opened specimens. The bottom sections of specimen halves show resultant cohesive fracture surfaces when specimens were split open after the 56 day exposure test period.

FIGURE 16. OPENED WEDGE SPECIMENS WITH PF-1 PHOSPHATE FLUORIDE PA MODIFIED PRETREATMENT AND BR127/FM300K ADHESIVE SYSTEM

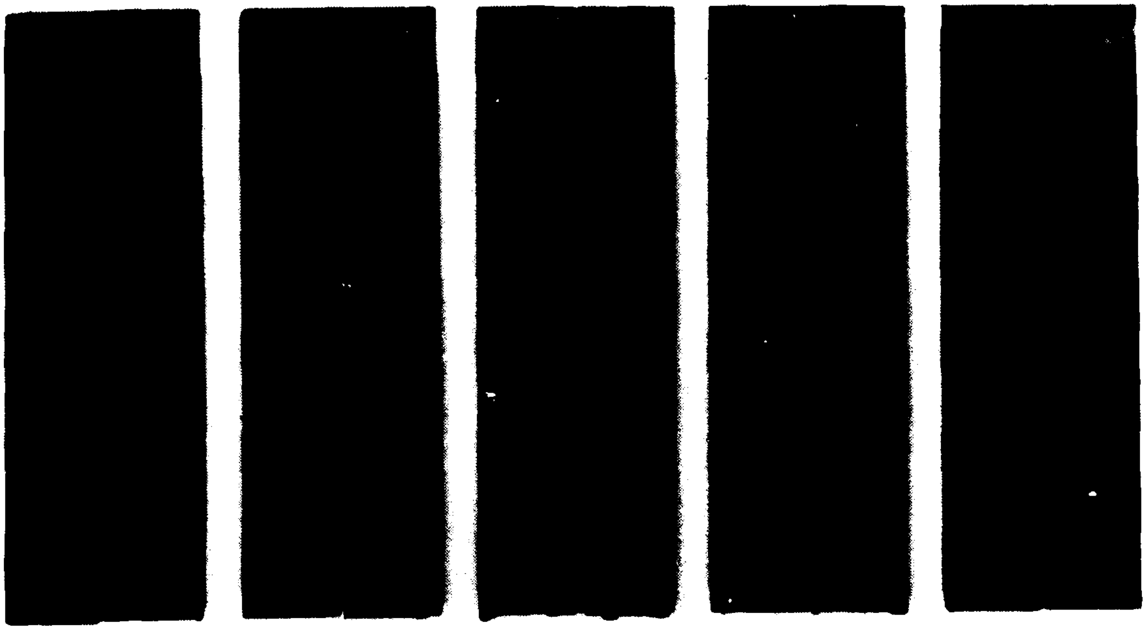


FIGURE 17. OPENED WEDGE SPECIMENS WITH DP-2 DRY HONE PASA JELL 107C PRETREATMENT AND BR127/FM300K ADHESIVE SYSTEM

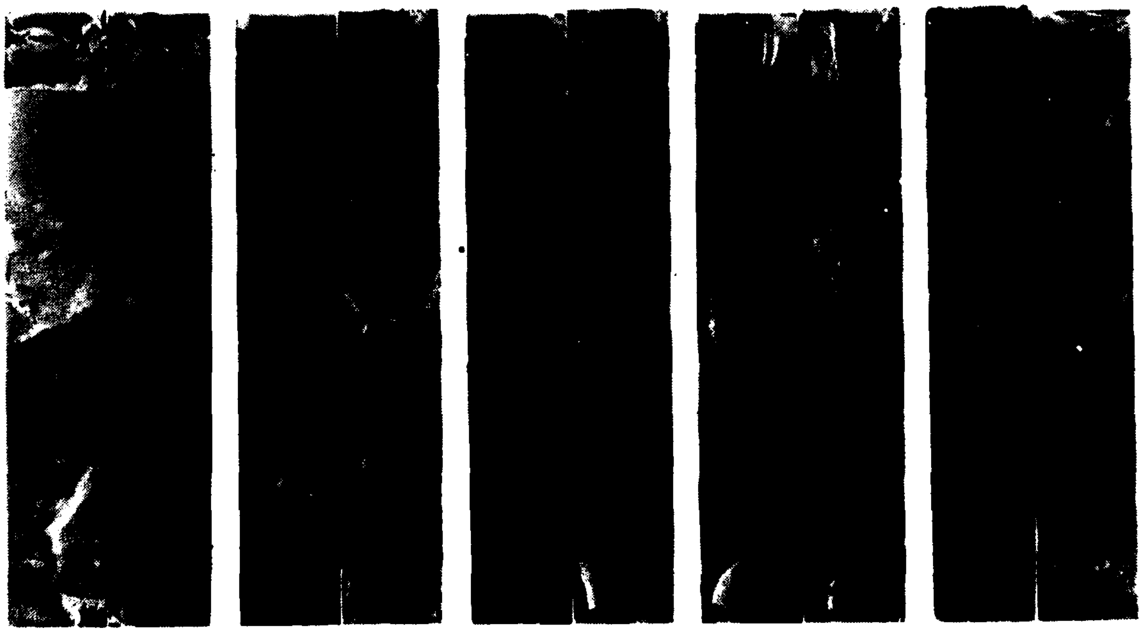


FIGURE 18. OPENED WEDGE SPECIMENS WITH PF-3 PHOSPHATE FLUORIDE/ HNO_3 (NITRIC ACID) PREDIP PRETREATMENT AND BR127/FM300K ADHESIVE SYSTEM

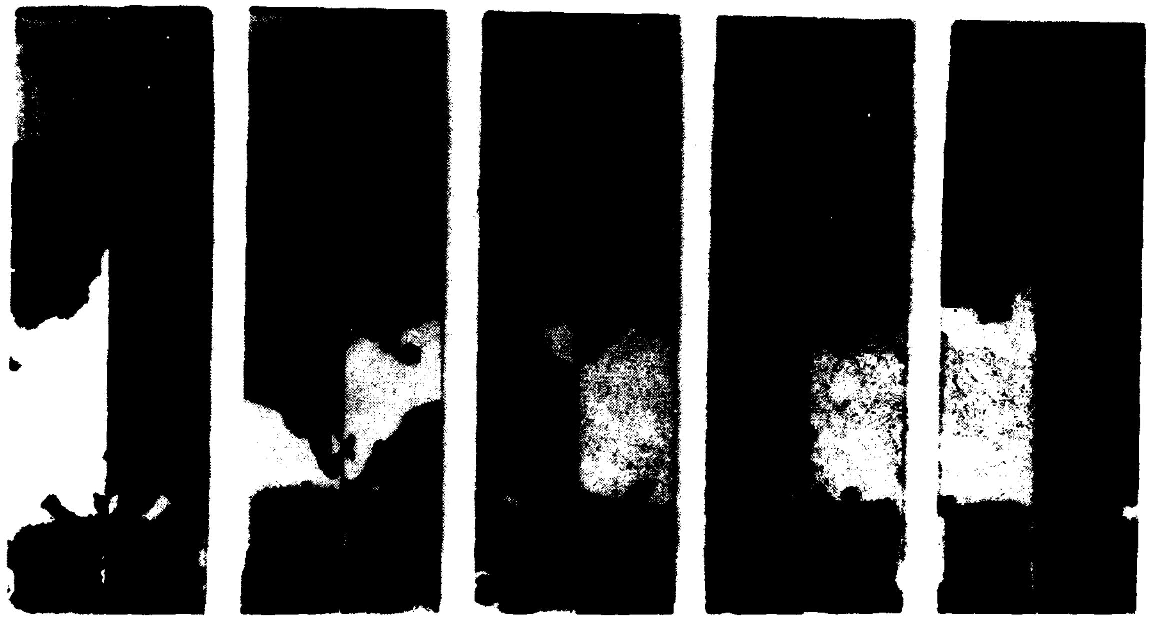


FIGURE 19. OPENED WEDGE SPECIMENS WITH PF-4 PHOSPHATE FLUORIDE PRETREATMENT AND BR127/FM300K ADHESIVE SYSTEM

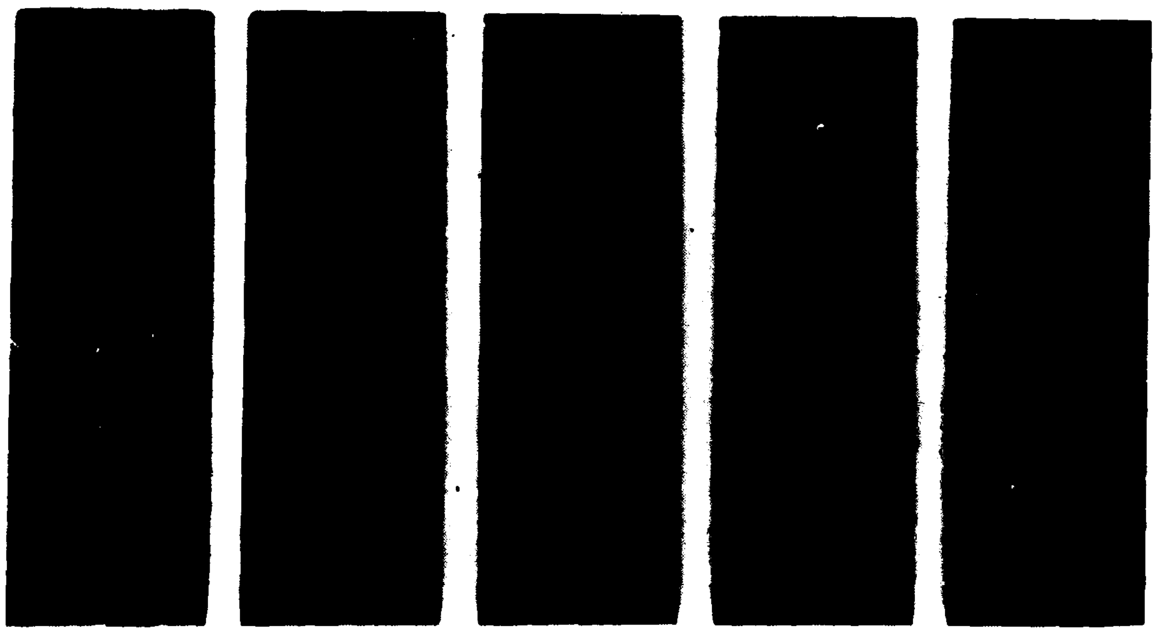


FIGURE 20. OPENED WEDGE SPECIMENS WITH CA5-4 CHROMIC ACID ANODIZE/FLUORIDE 5 VOLT PRETREATMENT AND BR127/FM300K ADHESIVE SYSTEM

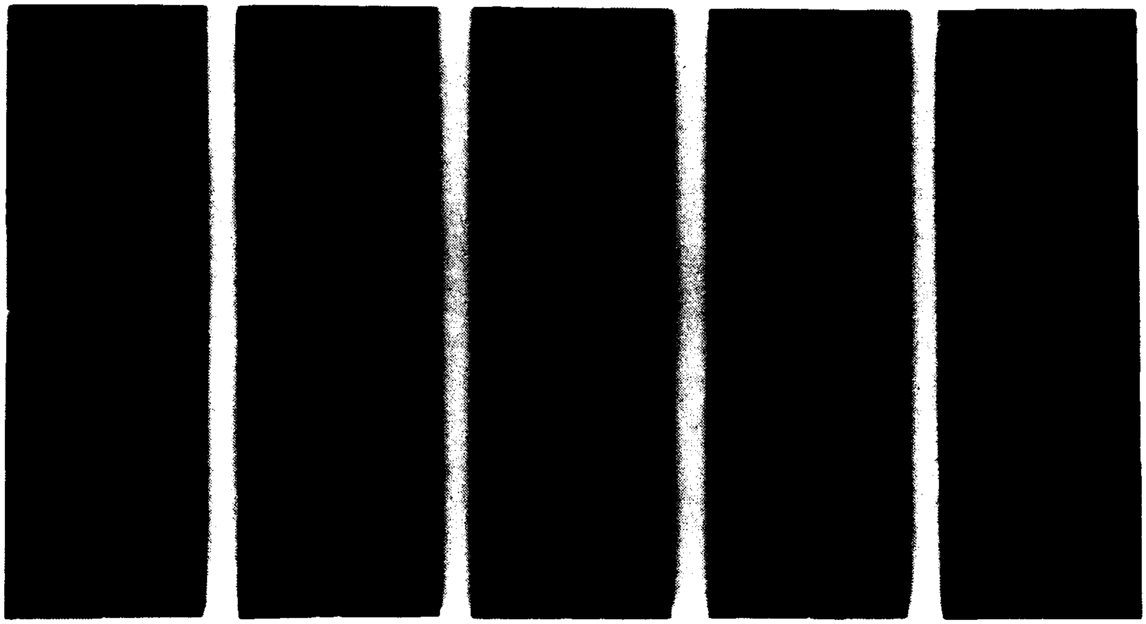


FIGURE 21. OPENED WEDGE SPECIMENS WITH CA10-4 CHROMIC ACID ANODIZE/FLUORIDE 10 VOLT PRETREATMENT AND BR127/FM300K ADHESIVE SYSTEM

FIGURE 22. OPENED WEDGE SPECIMENS WITH DA-5 DAPCOTREAT 4023/4000 PRETREATMENT AND BR127/FM300K ADHESIVE SYSTEM

FIGURE 23. OPENED WEDGE SPECIMENS WITH LP-6 PASA JELL 107C - WET HONE PRETREATMENT AND BR127/FM300K ADHESIVE SYSTEM

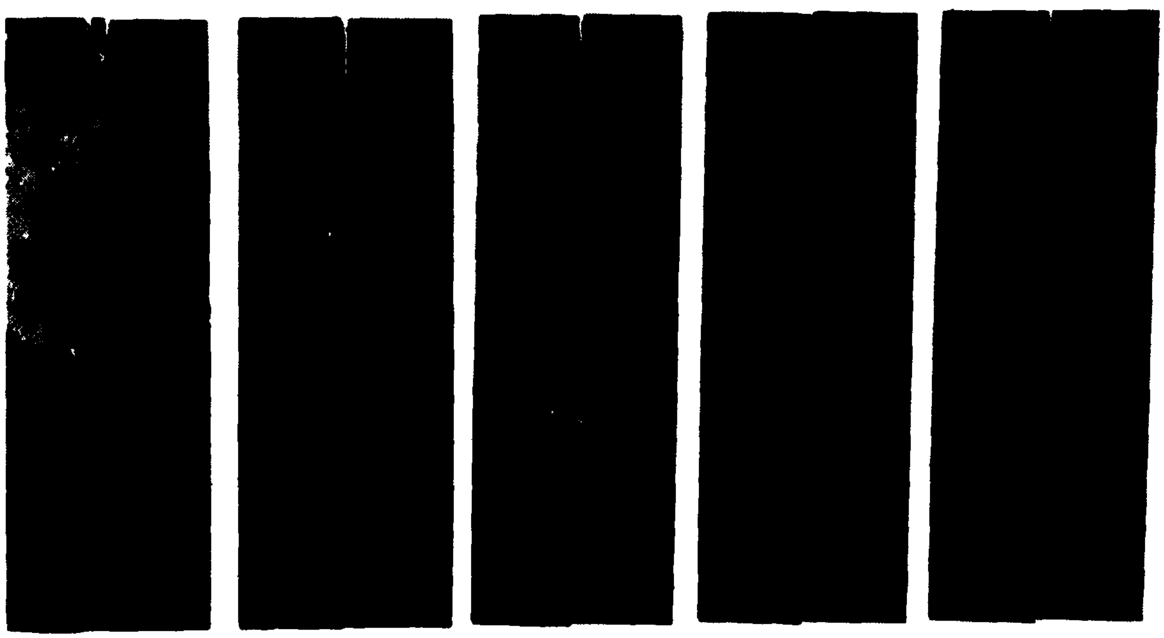


FIGURE 24. OPENED WEDGE SPECIMENS WITH VA-7 VAST PRETREATMENT AND BR127/FM300K ADHESIVE SYSTEM

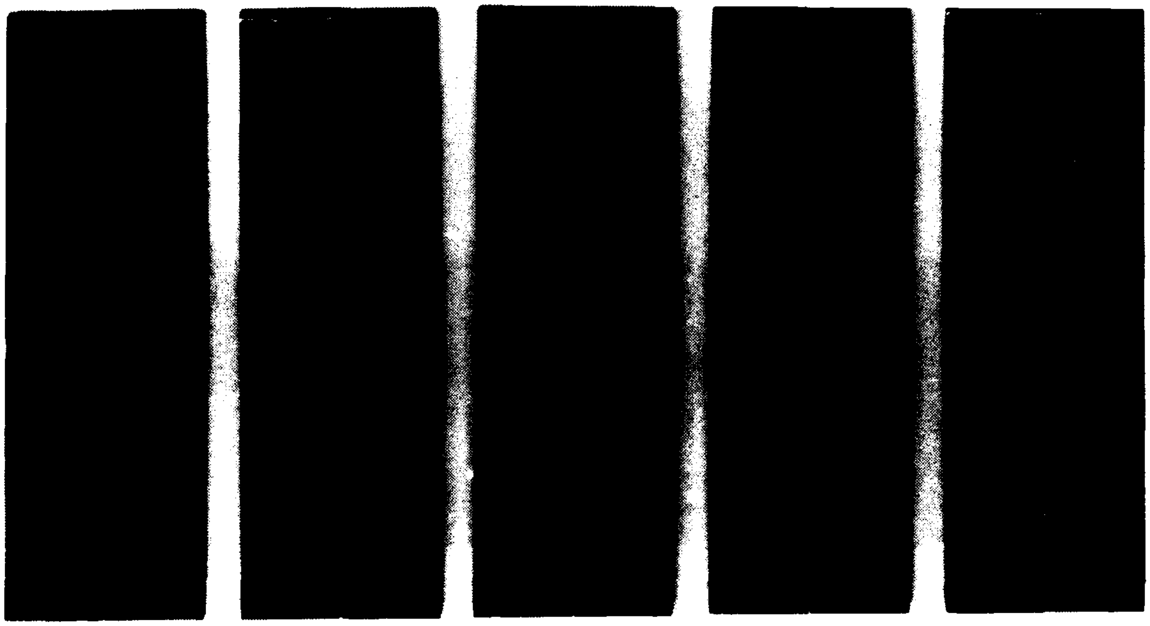


FIGURE 25. OPENED WEDGE SPECIMENS WITH TU-8 TURCO 5578 PRETREATMENT AND BR127/FM300K ADHESIVE SYSTEM

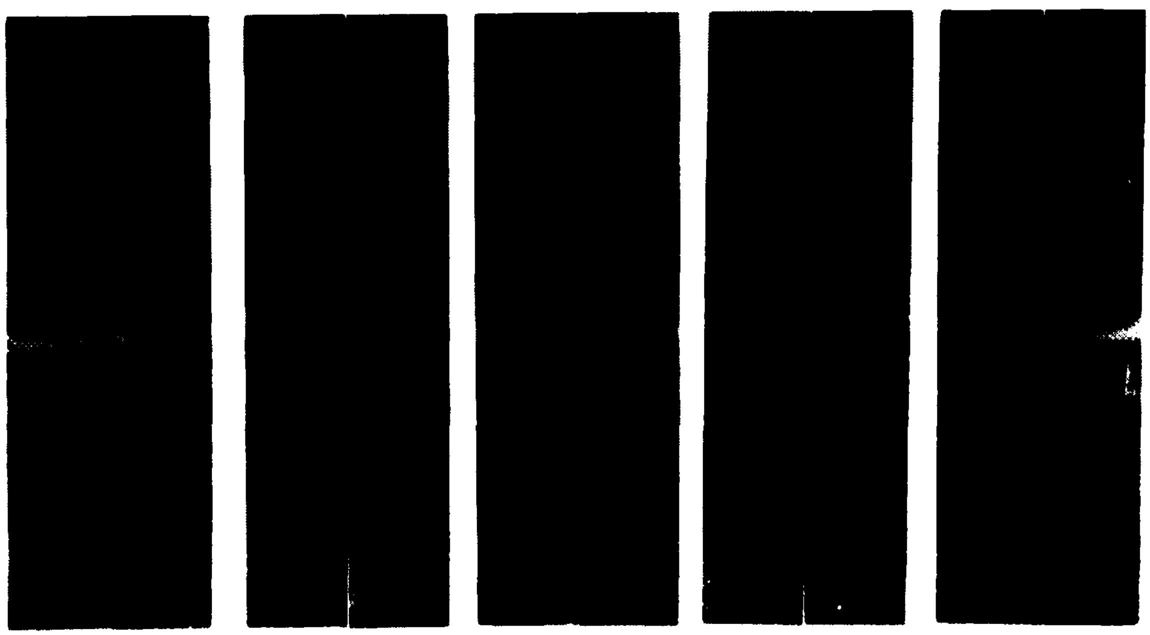


FIGURE 26. OPENED WEDGE SPECIMEN WITH AP-9 ALKALINE PEROXIDE PRETREATMENT AND BR127/FM300K ADHESIVE SYSTEM

Opened wedge specimens bonded with the other three adhesive systems are shown in Figures 27-29.

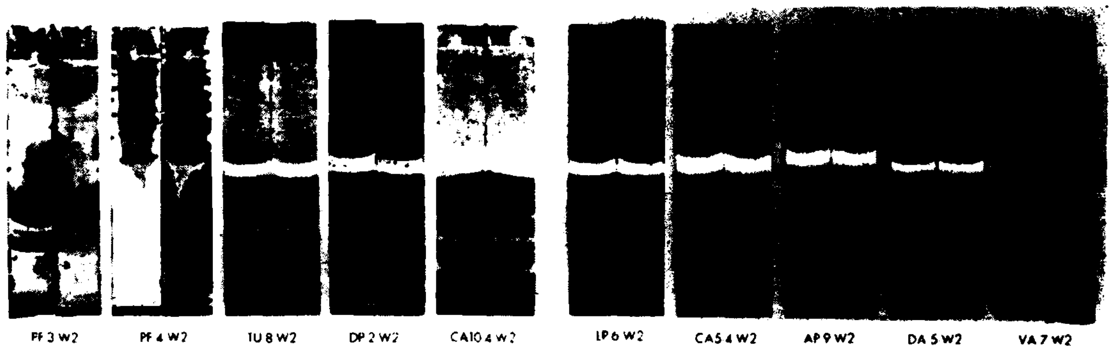


FIGURE 27. OPENED WEDGE SPECIMENS AFTER 56 DAYS EXPOSURE BR127/FM73M

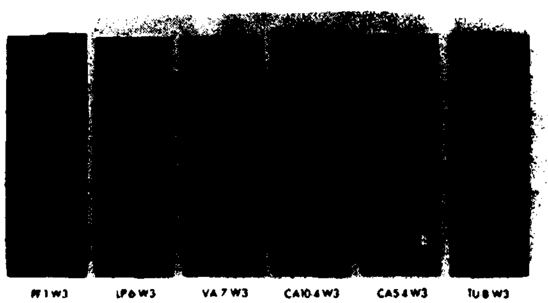


Figure 28A. Opened Wedge Specimens after 56 Days Exposure M329 Type II/M329

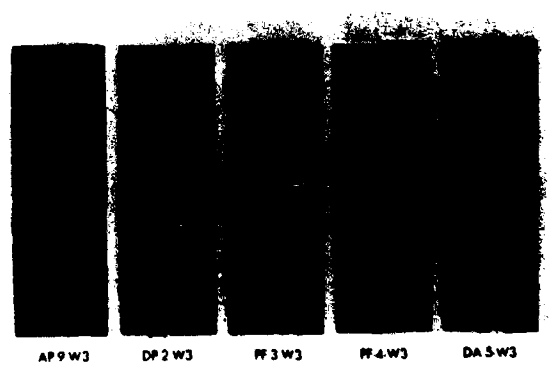
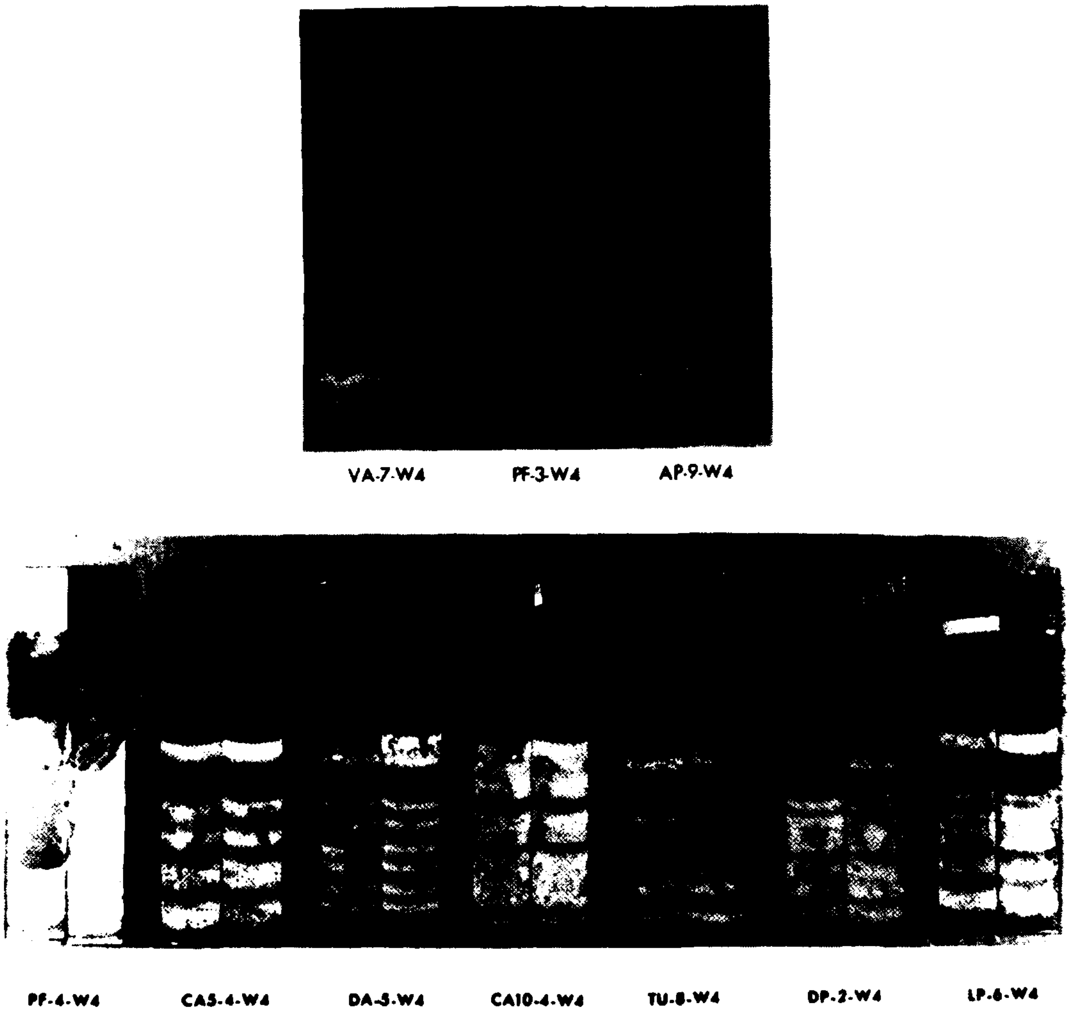


Figure 28B. Opened Wedge Specimens after 56 Days Exposure M329 Type II/M329



TITANIUM SURFACE TREATMENT TREATMENT PROGRAM
CRACK EXTENSION WEDGE TEST 140° F, 100% RH

FIGURE 29. OPENED WEDGE SPECIMENS AFTER 56 DAYS EXPOSURE BR127/EA9628H

The same general failure pattern is seen on all specimens regardless of pretreatment type or adhesive system. First is a cohesive failure zone caused by wedge insertion. Next is the crack growth zone which is predominately adhesive failure. The crack growth zone is more extensive and dramatic with the low durability pretreatments. Finally, the bottom portion of each specimen exhibits another cohesive failure zone caused by separating the specimen halves.

CONCLUSIONS

1. The most durable titanium pretreatment systems as determined by wedge crack testing were chromic acid anodize with fluoride (both 5 and 10 volt), Turco 5578 alkaline etch, liquid hone Jell 107C, and alkaline peroxide.

2. Dapcotreat 4000 and dry hone Pasa Jell 107M were slightly lower in overall performance than the above five pretreatments.

3. The three phosphate fluoride pretreatments along with the VAST pretreatment resulted in significantly longer crack growths and poorer durability than the other pretreatments.

4. When using wedge tests, total wedge crack openings (a+Δa) are preferred over crack growth lengths (Δa) as a measure of durability.

RECOMMENDATIONS

Pretreatment methods recommended for adhesive bonding of titanium are chromic acid anodize with fluoride, Turco 5578, Pasa Jell 107, alkaline peroxide or Dapcotreat 4000. Production experience is limited with the chromic acid anodize and alkaline peroxide processes. Alkaline peroxide solutions are unstable so stringent production controls must be used with this method. Do not use phosphate fluoride and VAST pretreatments for titanium when high durability adhesive bond joints are required.

FUTURE PLANS

The alkaline peroxide process will be further developed to improve both stability and determine effective operating ranges. The alkaline peroxide pretreatment process provides durable adhesive bonds without treatment by solutions containing either chromate or fluoride chemicals and does not require anodizing equipment. Titanium surfaces prepared by the alkaline peroxide process are relatively stable with some roughness for good mechanical bond strength. Solution temperatures up to 160 F are required, but this is below the 190-200 F temperature required for alkaline etch processing.

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(c) Naval Air Systems Command Contract Nos. N00019-79-C-0294, N00019-80-C-0508 and N00019-81-C-0355, Bondability of Titanium Adherends.

(d) Ripling, E. J., Mostovoy, S., and Carten, H. T., "Fracture Mechanics: A Tool for Evaluating Structural Adhesives," Journal of Adhesion, 1971, Vol. 3, pp 107-123.

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(h) Marceau, J. A., Moji, Y., and Mc Millan, J. C., "A Wedge Test for Evaluating Adhesive Bonded Surface Durability," Adhesives Age, Vol. 20, No. 10, October, 1977.

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